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The chemistry of the sugar cane and its products in Louisiana

Charles Albert Browne

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The Chemistry of the Sugar Cane and Its Products in Louisiana.

BY

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The Chemistry of the Sugar Cane and Its Products in Louisiana.

(By C. A. Browne, Jr., and R. E. Blouin.)

The data collected in this bulletin represent a summary of work performed during the past few years in the sugar house and laboratories of the Sugar Experiment Station of the Louisiana State University at Audubon Park, New Orleans.

I. COMPOSITION OF THE SUGAR CANE.

As is well known, the sugar cane in Louisiana, on account of the cool winters, seldom reaches maturity. It is only during an exceptionally mild winter that the cane arrows and produces seed, and this occurrence then is confined entirely to the extreme southern parishes of the State along the Gulf. The conditions of growth, and the methods of cultivating the cane crop in Louisiana are therefore necessarily very different from tropical countries, and as a result very marked differences are evident in the composition of the cane and its products between the two regions. It should also be remarked that within Louisiana itself, a great dissimilarity of conditions prevail, not only as regards climate, but also in the character of soils and processes of manufacture, so that there are perhaps wider local variations in composition than are noticeable in any other cane-producing country.

Table No. I shows the proximate composition of the leaves, stalks, roots and seed of the sugar cane, according to analyses by Halligan and Agee.

TABLE NO. I.

(Proximate Analysis of Sugar Cane.)

	Leaves.	Stalks.	Roots.	Seeds.
Water	74.38%	74.96%	68.79%	11.03%
Ash	2.23	0.64	1.87	5.22
Fat and Wax	0.69	0.38	0.54	2.01
Nitrogenous bodies, (protein, etc.)	1.70	0.58	1.59	8.47
Fiber — Crude cellulose	9.18	4.86	9.58	25.51
Fiber — Pentosans	5.49	3.04	7.04	26.26
Fiber — Ligneous bodies	4.13	2.14	4.25	21.50
Sugars, etc.	2 20	13.40	6.34	.....
Total	100.00	100.00	100.00	100.00

A somewhat closer inspection of the various constituents given in Table I will be of value, particularly as regards the composition of the cane stalk.

Water. The water content of the sugar cane is somewhat variable, decreasing as the period of maturity advances and also depending upon the wetness or dryness of the season. Canes after being cut lose water rapidly from evaporation, with a corresponding increase in the per cent of solids in the juice. Analyses of juices from canes that have been long windrowed or shipped a considerable distance frequently lead to erroneous conclusions regarding sugar content.

Ash. The percentage composition of the ash of the sugar-cane in Louisiana is found to vary widely according to the variety of the cane, type of soil and manner of fertilization. Analyses made by Hall of the ash from the leaves. stalks and roots of the Demerara No. 74 cane are given in Table II.

TABLE II.

(Composition of Ash of Sugar Cane.)

		Ash of Leaves.	Ash of Stalk.	Ash of Roots.
Potash	K_2O	31.25%	38.23%	17.39
Soda	Na_2O	1.17	1.30	0.85
Lime	CaO	5.90	5.19	3.45
Magnesia	Mg O	5.11	5.76	2.61
Iron Oxide	Fe_2O_3	1.45	1.13	3.60
Alumina	Al_2O_3	1.03	0.25	4.70
Silica	$Si O_2$	30.32	15.70	49.52
Phosphoric Acid	P_2O_5	7.25	5.27	3.99
Sulphuric Acid	$S O_3$	11.29	18.47	9.15
Carbonic Acid	CO_2	1.10	2.70	0.45
Chlorine	Cl	3.08	4.55	0.98
Carbon	C	0.16	0.54	2.30
Total		99.09	99.06	98.99
Deduct 0=Cl		0.70	1.02	.22
		98 39	98.04	98.77

Fat and Wax. The fat or oil of the cane is very small in amount and is confined mostly to the interior tissues, the pith and fibro-vascular bundles. The wax of the cane is found entirely upon the outer surface of the stalk and constitutes about 1 per cent of the rind. It is easily recognized by the white powdery coating which it gives the stalk, particularly in the region of the node. The properties of cane wax were first studied by Avequin, an apothecary of New Orleans, over sixty years ago.

He named the substance *cerosin* (from the Greek ceros, meaning wax) and gives the following description of its properties: "It is yellowish, very hard, easily pulverized to a white powder, and when moulded in the form of a candle burns like wax or spermaceti. It melts at 82 degrees C, solidifying again at 80 degrees. Its specific gravity is 0.961 at 10 degrees. It is odorless, unites with alkalies only with difficulty and does not change on exposure to the air." Avequin by scraping obtained more than two grams of wax from a purple stalk of cane. He also showed that an appreciable quantity of wax escaped into the juice during milling, the amount of this, however, being less than 1-100th of a per cent of the weight of juice.

Dumas, who made a chemical study of Avequin's cane wax, found it to contain 81.00 per cent carbon, 14.16 per cent hydrogen and 4.84 per cent oxygen, and regarded the substance as an alcohol of the formula C_{24} H_{50} O. Lewey repeated Dumas' work four years later at the latter's request and obtained for cane wax 81.74 per cent carbon, 13.64 per cent hydrogen and 4.62 per cent oxygen and assigned the formula C_{24} H_{48} O.

Nitrogenous Bodies. The total percentage of nitrogen in the sugar cane is relatively small, the average of many analyses made at this station being only about .05 per cent. This small amount of nitrogen is distributed among a large number of different bodies, each one of which plays an important part in the physiological processes of the cane. The distribution of the nitrogen among the different constituents of the cane can be seen from the following analyses, which were made upon several stalks of the Louisiana Purple variety:

TABLE III.

	Percentage of Cane.
Albumen (coagulable and soluble in pepsin)	0.059
Nucleins, etc. (coagulable, but insoluble in pepsin)	0.040
Albumoses and peptones (not coagulable)	0.033
Amido Acids (Aspartic acid)	0.145
Amido Acid Amids (Asparagin)	0.232
Ammonia NH_3	0.008
Nitric Acid, N_2 O_5	0.071
Total nitrogenous bodies	0.588

The above percentages are subject to considerable variations according to the age and variety of the cane, manner of fertilization and cultivation. The nitrogenous bodies are not distributed evenly throughout the stalk, the work of Beeson having shown a greater localization of albuminoids in the nodes and of amids in the internodes.

	Nitrogen. Albuminoid	Nitrogen. Amid	Nitrogen. Total
Nodes	0.1778	0.0051	0.1829
Internodes	0.0559	0.0258	0.0817

Fiber. The fiber of the sugar cane is distributed among the three principal tissues, the rind or shell, the pith, and fibro-vascular bundles. A mechanical separation of these tissues of the cane from one another gave the following percentage composition. The analyses were performed upon a mature stalk of the Louisiana Purple cane.

	Pith Per cent.	Bundles Per cent.	Rind Per cent.
Whole cane (3 analyses)	2.39	1.81	5.51
Dry fiber	24.66	18.60	56.74

A proximate analysis of the above tissues is given below. The results were calculated to a moisture-free basis.

TABLE IV.

	Pith Per cent.	Bundles Per cent.	Rind Per cent.
Ash	1.68	3.58	1.64
Fat and wax	0.41	0.72	0.98
Protein	1.94	2.00	2.19
Cellulose (method of Cross and Bevan)	49.00	50.00	51.00
Pentosans (furfuroids)	32.04	28.67	26.93
Lignin (by difference)	14.93	15.03	17.17

With the exception of ash the results for the different tissues show a certain regularity, the bundles standing intermediary between the pith and the rind. The analytical data shows that we have in the pith a minimum and in the rind a maximum degree of lignification.

The fiber is not distributed evenly throughout the cane, the contents of woody matter being twice as high in the region of the nodes. Beeson, who has made a special study of this point, gives the following analyses. (Bull. 38, La. Sugar Expt. Station, p. 1353.)

Top	Node	15.86%
	Internode	8.60
Middles	Node	15.90
	Internode	8.00
Butt	Node	18.20
	Internode	8.08

A study of the hydrolytic products obtained by digesting purified bagasse with caustic soda showed cane fiber to be an exceedingly complex substance. The following results calculated to 100 parts of cane-fiber (protein, ash, fat, etc., excluded) give the approximate percentage of the different hydrolytic products.

	Per cent.
Cellulose (including oxycellulose)	55
Xylan	20
Araban	4
Lignin	15
Acetic Acid	6

The *cellulose* $(C_6 H_{10} O_5)n$, obtained from the sugar cane resembles that obtained from corn stalks in many of its properties. The pith cellulose is very easily attacked by concentrated alkalies and for this reason great care must be exercised in manufacturing paper-stock from bagasse.

The *Pentosans,* xylan and araban $(C_5 H_8 O_4)n$ constitute the cane gum. These constituents of the fiber are easily soluble in alkalies, from which they are precipitated by alcohol as a gummy deposit. Inversion of the cane gum with hydrochloric acid produced the pentose sugars, xylose and arabinose, $(C_5 H_{10} O_5)$, which on removal of the acid, were easily obtained in the crystalline form. The specific rotation of xylose obtained from cane gum was found to be +18.5 and of arabinose +104.2.

The *Lignin* is obtained from cane fiber by digesting with solutions of alkalies to which it imparts a yellowish brown coloration. After removing the pentosans with alcohol, the lignin can be precipitated by evaporating the filtrate and adding a slight

excess of acid. It constitutes a resinous deposit easily reduced to a yellowish powder, and soluble in alcohol and alkali solution. The elementary composition of the purified lignin obtained from cane fiber was found to be:

	Found. I	Found. II	Theoretical for $C_{24} H_{26} (CH_3)_2 O_{10}$
Carbon	61.89%	61.75%	61.90%
Hydrogen	6.17	6.14	6.35

The constitution of cane lignin agrees with the formula proposed by Lindsey and Tollens for wood lignin given above.

Acetic Acid, CH_3 COOH, the well known acid of vinegar, may be obtained from cane fiber by digesting with caustic alkalies and then distilling with a slight excess of sulphuric acid.

The above substances do not exist in cane fiber as a mechanical mixture, but in a state of most intimate combination, forming a very complex molecule whose exact structure is not yet understood.

The Sugars. The three principal sugars of the cane are sucrose, dextrose and levulose.

Sucrose, ($C_{12} H_{22} O_{11}$), is the constituent for which the cane is most prized and its physical properties are too well known to require mention. Its solutions rotate the plane of polarized light to the right, the specific rotation being +66.5. By means of inverting agents sucrose is split up into equal parts of dextrose and levulose, hence the name of the mixture—invert sugar.

Dextrose, ($C_6 H_{12} O_6$), sometimes known as grape sugar, occurs in all parts of the sugar cane. It is a white crystalline body, easily soluble in water, the solution rotating to the right. Specific rotation +53.

Levulose, ($C_6 H_{12} O_6$), sometimes known as fructose or fruit sugar, occurs associated with dextrose in all parts of the sugar cane. It is easily soluble in water and much less easily crystallized than dextrose; solutions of levulose rotate strongly to the left, the specific rotation at 20 degrees C being —89.2. Both dextrose and levulose, like other simple sugars, exert a strong reducing action upon alkaline copper and silver solutions, and from this property are often termed reducing sugars. In commercial work dextrose, levulose and other reducing bodies are

generally comprised under the one term glucose, although chemically speaking the word glucose is only applicable to dextrose.

The pentose sugars, xylose and arabinose, seem to occur in traces in decomposed canes, the result no doubt of the inversion of the xylan and araban contained in the fiber. The presence of maltose and raffinose has also been reported in the sugar cane, but this requires confirmation. The optically inactive sugar of the cane, anoptose, which has been reported, is probably only an inactive mixture of dextrose and levulose.

Among other ingredients of the cane which have not been mentioned are the Pectins or Gums, and the Acids.

Pectins or Gums. The sugar cane contains a small amount of pectinous or gummy matter, which escapes into the juice during the milling. These gums are soluble, but are thrown out as a flocculent precipitate on adding alcohol to the concentrated juice. The quantity of soluble gums is much higher in some varieties of cane than in others. The gums of the juice are derived entirely from the hemicellulose of the cane fiber and are composed of xylan and araban, with some galactan, the latter being more evident in unripe canes than in canes which have reached maturity.

Acids. The acids of the cane exist partly in the free condition, the larger amount, however, occurs combined with potash and other bases in the form of salts. On incineration these organic salts are destroyed, the basic constituents being left in the ash as carbonates. Among the acids of the sugar cane are aspartic acid (already mentioned among the nitrogenous bodies), malic acid, and succinic acid, all closely related to one another constitutionally and physiologically. Glycolic acid also occurs in green canes. Tannic acid is always present, especially in the peripheral region and in the growing parts. The tissues near the buds and eyes of the cane always give a strong reaction for tannin bodies. Citric, tartaric and aconitic acids have also been reported in the cane, but their presence requires further confirmation.

Among other constituents of the cane which have not been enumerated, should be mentioned the coloring matter. The chlorophyll which gives the leaf of the cane its green color is also present in very small amount in the stalk, though its pres-

ence there is often marked by a red or purple coloring-matter (anthocyan), as in the Purple, Striped and other colored canes.

Starch is another ingredient which occurs in the leaf of the cane and in traces in the green portions of the stalk. In the mature joints, however, starch is almost if not completely absent. The relationship of starch to the sugars of the cane will be discussed under the next section.

II. THE PHYSIOLOGY OF THE GROWTH AND RIPENING OF SUGAR CANE.

During germination and for the first few weeks of its growth the young sugar cane is entirely dependent for its supply of plant food upon the mother cane. This food supply is largely made up of sugars and nitrogenous material. The sucrose of the mother cane undergoes a slow inversion, as is always the case in plants when this reserve material is to be transported to points of growth. Juice from a sound mother cane that had lain in the ground two years at Audubon Park contained 1.40 per cent of sucrose and 0.91 per cent of reducing sugars, so it will be seen that the process of inversion is one of long duration. This inversion of sucrose goes on more rapidly in the region of the bud and is due to the activity of an inverting ferment or enzyme developed during the process of germination.

In addition to the inversion of sucrose, the albuminoids of the mother cane undergo a transformation, being first changed by enzymes to albumoses or peptones and then still further reduced to asparagin and aspartic acid in which form they are transported to the young plant. The work of Beeson, previously referred to, shows that the quantity of albuminoid material is three times greater in the nodes than in the internodes, which circumstance is clearly a provision of the mother cane for the needs of the growing bud. The presence of considerable asparagin in the young suckers of sugar cane was demonstrated by Maxwell. The asparagin and other amid bodies derived from the albuminoids of the mother cane are again transformed in the young plant to albumen, which is the principal constituent of the cell protoplasm. The sugars are changed to cellulose and hemicellulose, and go to build up the cell walls and tissues of the growing plant.

As soon as the young cane has developed leaves and roots it gradually ceases to be dependent upon the mother cane and is in position to shift for itself. The process of assimilation now begins; the carbonic acid of the air and the water taken up through the roots undergo a transformation in the chlorophyll bearing tissues of the leaf, with the result that starch and also probably sugar are formed. The presence of starch in cane leaves can be very easily demonstrated by dissolving the chlorophyll in boiling alcohol and then applying tincture of iodine, when the starch grains will be colored blue. In this process of assimilation oxygen is set free, as is shown by the following formula:

Carbonic acid.	Water.	Starch (sugar).	Oxygen.
$12\ CO_2$	$+12H_2O=$	$C_{12}\ H_{20}\ O_{10}\ (2H_2O)+$	$12O_2$

The process of assimilation can only go on in the sunlight; it ceases during the night and is of course less active on cloudy days than when the sky is clear.

Parallel with the formation of starch and sugar in the leaf is the formation of albuminoid matter. The nitrates and sulphates of the soil are taken up by the roots, together with the other mineral constituents and are transported in solution to the leaf, where a combination of the nitrogen and sulphur with the sugars is effected with the formation of albumen. The exact nature of this change, which is of an exceedingly complex character, is not yet understood. Hofmeister's formula for albumen is $C_{450}\ H_{720}\ N_{116}\ S_6\ O_{140}$, and such a compound would require 75 molecules of dextrose, 116 of potassium nitrate and 6 of calcium sulphate for its formation.

In addition to the starch which is formed in the leaf of the cane a considerable amount of sucrose is formed. This sucrose may be formed directly in the process of assimilation or it may be derived secondarily from the starch. But neither the starch nor the sucrose remain as such in the leaf for any length of time, both being converted by enzymes to reducing sugars and then transported to points of growth where they are utilized in the building up of new tissue. This process of conversion goes on at night as well as by day, the assimilative products which accumulate by day being in large part removed during the night.

These and other changes which go on in the young cane can best be illustrated by the following analyses made upon the juice from the leaves of a young cane at night and at morning, and from different parts of the stalk of the same:

TABLE V.

	Evening. Per cent.	Morning. Per cent.
Brix	5.77	5.09
Sucrose	0.94	0.54
Dextrose	0.63	0.86
Levulose	0.69	0.77
Ash	1.42	1.24
Free Acid	0.27	0.27
Combined Acid	0.62	0.54
Nitrogenous bodies	0.15	0.18
Gums	0.30	0.17

We notice in the juice of the leaves a large decrease in the amount of sucrose during the night with a corresponding increase in the amount of reducing sugars. In the top joints we note an accumulation of the reducing sugars brought down from the leaves; at this point, which is the region of most intense growth, we have the greatest disparity between sucrose and reducing sugars, the glucose ratio being 461.84. In the middle joints where the process of growth is being suspended, we observe that the reducing sugars are being reconverted to sucrose, which is henceforth stored up in the pith cells as reserve material. The bottom joints illustrate the same facts only to a greater degree. Regarding the other constituents it will be seen that the percentages of ash, free and combined acid, nitrogenous bodies, and gums all decrease as we pass from the leaves down the stalk.

The same phenomena may be observed if we compare the analyses of whole canes made at different periods of their growth. In the following table analyses are given of D.74 and D.95 canes at various intervals between the middle of July and the middle of October.

TABLE VI.

(Analyses by Halligan and Verret.)

Variety.	Ingredient.	July 19.	Aug. 2.	Aug, 17.	Sept. 1.	Oct. 3.	Oct. 17.
D. 74	Weight stalk	544 gm.	584 gm.	916 gm.	1364 gm.	1431 gm.	1243 gm.
	Fiber	3.84 %	4.32 %	6.36 %	.....	7.96 %	8.18 %
	Sucrose	1.50	2.14	4.88	6.07 %	8.59	11.04
	Dextrose	1.72	1.75	4.75	1.72	1.85	1.05
Stubble	Levulose	1.69	1.64	1.49	1.38	1.31	.87
	Ash	.57	.49	.48	.49	.42	.43
	Acids	.36	.25	.22	.22	.20	.18
	Albuminoids	.10	.09	.08	.06	.07	.08
	Amids	.08	.04	.06	.04	.03	.04
	Gums	.19	.17	.13	.16	.07	.09
D. 95		July 29.	Aug. 15.	Aug. 26.	Sept. 7.	Oct. 3.	Oct. 17.
	Weight stalk	466 gm.	651 gm.	854 gm.	1050 gm.	1375 gm.	1116 gm.
Stubble	Fiber	.....	5.10 %	5.59 %	6.57 %	7.82 %	8.10 %
	Sucrose	1.23 %	2.45	3.02	4.43	8.19	8.92
	Dextrose	1.92	2.06	2.37	1.98	2.22	1.46
	Levulose	1.81	1.81	1.98	1.94	1.55	1.37
	Ash	.46	.41	.29	.34	.32	.38
	Acids	.23	.22	.17	.17	.12	.13
	Albuminoids	.11	.09	.08	.07	.06	.08
	Amids	.06	.05	.02	.03	.03	.04
	Gums	.10	.05	.05	.07	.04	.07

Pressure of other work prevented the analyses being carried through to the complete maturity of the canes, yet the results are sufficient to illustrate the general character of the changes during the period of growth. We note a regular increase in the percentage of fiber and sucrose, although the increase of these two ingredients does not go on *pari passu* as has been sometimes claimed. Attempts to establish a fixed ratio between fiber content and sucrose content are absolutely futile. The percentages of ash, acids, nitrogenous bodies and gums all show a decrease as the cane matures.

The relations of the sugars of the cane to one another during the period of growth and their effect upon the polarization of the juice is a subject of more than theoretical importance to the sugar planter.

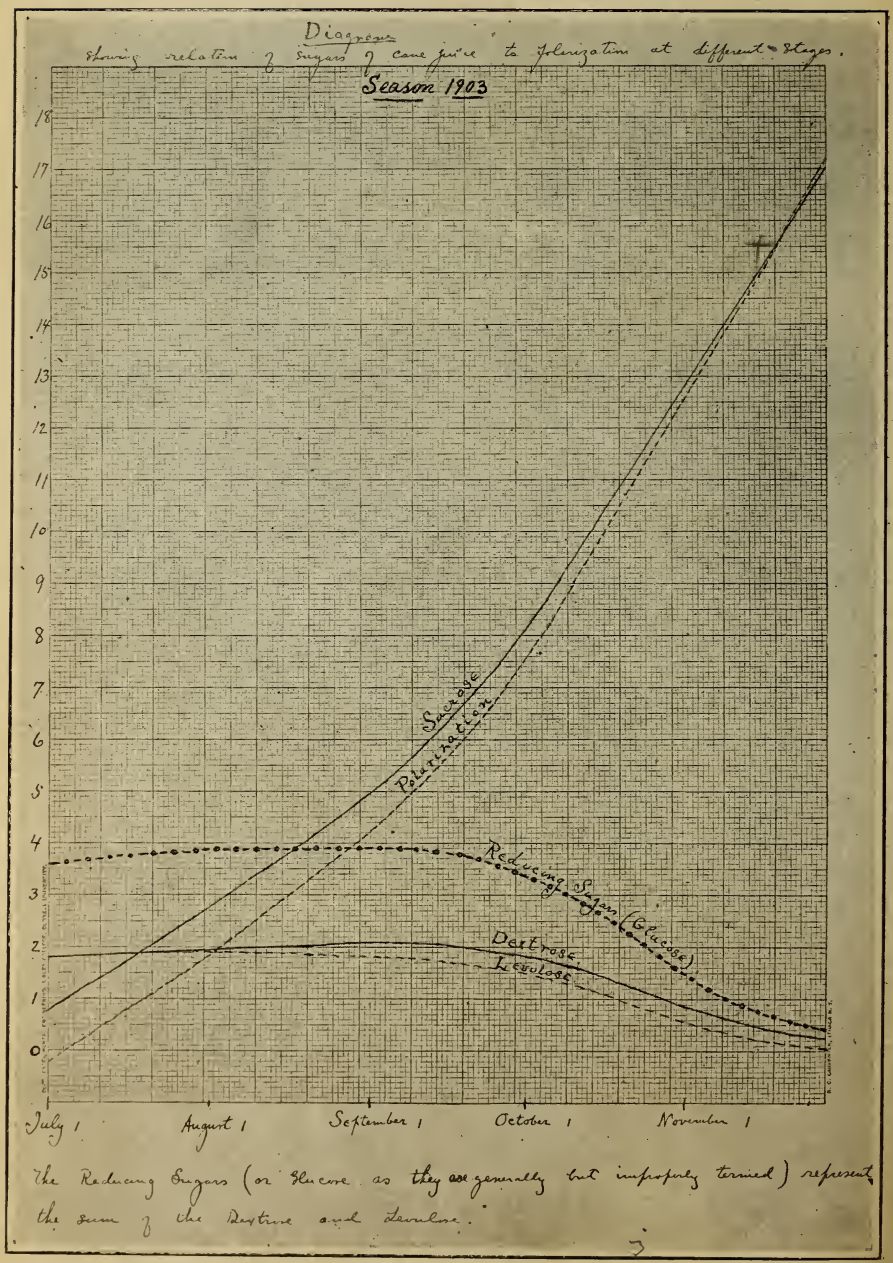
It will be observed from the analyses in Tables V and VI that the reducing sugars, dextrose and levulose, in the green tops and joints of the cane and also in the stalks of very young canes, are present in very nearly equal amounts; in other words, they are present as invert sugar, being derived, as we have seen, by the inversion of the sucrose in the leaf. In the riper joints of the cane, however, this relation no longer exists. A considerable quantity of the invert sugar is reconverted into sucrose, but another portion is consumed in the vital processes of the cane (formation of new tissue, etc.); for the latter purpose levulose appears to be used in greater amount than dextrose with the result that the latter sugar is left in excess. The disparity in the percentages of dextrose and levulose increases as the cane matures until finally the levulose may nearly disappear, as in the following analysis of the juice from T.111 cane made November 28, 1903, at Audubon Park.

Brix	19.45
Sucrose	18.29
Dextrose	0.21
Levulose	0.04

The above condition of maturity was due to exceptional climatic conditions during 1903, and is very rarely attained in Louisiana. In tropical countries, however, where complete maturity of the cane is attained the levulose of the sugar cane may

disappear. (Went. Chem. Physiol. Untersuch. des Zuckerrohrs Jahrbuch. für wissenschaft. Botanik XXXI, p. 289.)

These fluctuations in the percentages of the different sugars naturally affect the polarization of the juice to a marked degree. The relation between the sugars and polarization of cane juice at different periods of growth of the Purple cane in 1903 is shown in the following diagram:



The Reducing Sugars (or Glucose as they are generally but improperly termed) represent the sum of the Dextrose and Levulose.

It will be noted that at very early stages of growth the juice shows a minus reading due to the preponderance of invert sugar; somewhat later the juice becomes inactive, showing no rotation whatever, though double polarization shows about 1 per cent of sucrose. Then the rotation becomes a positive quantity and as the cane matures gradually approaches the true percentage of sucrose until finally the polariscopic reading and true percentage of sucrose coincide. At the latter stage the polarizing powers of dextrose and levulose neutralize one another. If conditions are favorable for complete ripening the levulose will nearly disappear. The rotary power of dextrose then predominates with the result that the polariscopic reading slightly exceeds the true percentage of sucrose. The latter condition, however, very rarely prevails in Louisiana. Under very exceptional climatic conditions the reducing sugars of the cane have been reported to disappear completely, in which case the polarization and sucrose content will again coincide. A case of the latter kind was reported in 1903 by Dr. H. W. Wiley upon canes grown in Florida.

III. PHYSIOLOGICAL ROLE OF THE ENZYMES OF THE SUGAR CANE.

Reference has already been made to certain nitrogenous ferments or enzymes which occur within the cane. Though present in but exceedingly minute amounts these ferments play a very important role in the physiological processes of the plant and require more than a passing mention.

If the green tops of a sugar cane be well macerated, the juice expressed, and treated with an antiseptic agent, such as chloroform or thymol to prevent fermentation by yeasts or bacteria, it will be found that the sucrose content of the juice undergoes a gradual diminution, though no traces of microorganic life are evident, and that simultaneously with this decrease in sucrose the content of reducing sugars increases. We have here a well marked instance of the activity of the enzyme *invertase,* the presence of which was noted in the leaf of the cane; this enzyme occurs almost universally throughout the vegetable kingdom, especially in the green or growing parts of plants. This presence of invertase has a very practical bearing outside of its physiological importance. The gradual falling off

in sucrose content of sugar cane which has been windrowed for any length of time is due very largely to spontaneous inversion. If the green tops of the cane are removed at the time of cutting, the loss of sucrose is much less evident. This can be easily seen in the following series of experiments which were carried out at Audubon Park in 1893. Several lots of cane were windrowed, one-half of each lot having the tops removed, and the other half retaining them. In all other respects the conditions of the experiments were perfectly similar. At the end of a month all tops were removed, the stalks from the different lots ground and the juices analyzed with the following results:

TABLE VII.

	Brix.	Sucrose.	Glucose.
Lot 1—Windrowed with tops cut.....	16.1	13.3	1.25
Windrowed with tops on.....	15.9	12.1	1.85
Lot 2—Windrowed with tops cut.....	15.8	12.8	1.22
Windrowed with tops on.....	15.4	11.5	1.53
Lot 3—Windrowed with tops cut.....	16.3	13.5	1.25
Windrowed with tops on.....	16.1	12.6	1.92
Lot 4—Windrowed with tops cut.....	16.2	13.7	1.00
Windrowed with tops on.....	15.8	11.8	1.85
Lot 5—Windrowed with tops cut.....	15.9	12.8	1.39
Windrowed with tops on.....	15.0	10.7	2.17

The loss of sucrose due to spontaneous inversion is very evident and this we can attribute very largely to the diffusion of the inverting enzyme from the green tops into the stalk. The inversion of sucrose naturally causes an increase in the glucose content, though this increase, it will be noted, is not proportioned to the loss in sucrose. This discrepancy is probably due to a destruction of the glucose from respiration in the leaf. The experiments show conclusively that the vital processes of the cane go on even after it is cut. It should not be forgotten that there is also a slow inversion of sucrose in canes that are windrowed with the green tops removed, though this inversion is attended by the concentration which the solids of the juice undergo from evaporation.

A very marked peculiarity of sugar cane juice, as of all vegetable juices, is the rapid darkening in color which takes

place immediately after expression. This darkening is much more evident within the body of the cane, especially in the region of the eyes and growing parts, when its tissues are laid open to the air. We have here the evidence of another enzyme belonging to the class of oxydases. The intense blue coloration which the tissues and juices of plants take on with tincture of guaiac is ascribed to an oxydase; the decomposing action which plant extracts exercise upon hydrogen peroxide discovered by Schoenbein, has been similarly explained, though Loew attributes the latter phenomenon to a special enzyme, catalase, and Pozzi Escot to a new class of ferments called reductases. Juice from sterilized cane exhibits none of the reactions named.

If certain polyphenols, such as hydroquinon, or pyrogallol, are added to fresh cane juice, a rapid oxidation of these compounds is produced with an intense darkening of the juice. The latter takes on at the same time a peculiar odor, due probably to the formation of a quinone body, and what is more remarkable, acquires a germicidal property which, in the case of the hydroquinone treated juice, insures its preservation for weeks. Sterilized juice shows no change in color and develops no germicidal properties with any of the phenol bodies named.

The darkening of vegetable tissues on their exposure to the air, has been explained by Bertrand to be due to the action of an oxidizing enzyme upon various tannin bodies, all more or less related to the polyphenols, and the query naturally arises, does cane juice itself exercise any germicidal properties in connection with the natural phenomenon of darkening? The conclusion which I have reached in investigating this point is that cane juice does acquire for a time such germicidal characteristics. Counting the bacteria in the expressed juice of the cane at regular periods usually shows for several hours a uniform decrease in numbers; with juice from sterilized canes, on the other hand, the bacterial content increases from the very start.* But it is especially within the body of the cane itself that this germicidal action is most evident, as when the cellular tissues are bruised or otherwise laid open to the air, and this we

*NOTE.—In this connection it is interesting to recall the vast amount of work done by Hunziker and others upon the germicidal action of fresh milk, and it may be that certain enzymes which we know to be always present in milk, exercise also here a toxic action.

might expect not only from the colloidal and adherent character of the enzymes, which renders them resistant to expression, but from the fact of localization which will be discussed later.

If we take two stalks of sugar cane, one raw and one sterilized, and puncture them with a knife; we will observe at the end of a few days a marked difference in the character of the wounds. The surface of the wound in the raw cane will be discolored, but free from the evidence of fermentation; the surface of the wound in the sterilized cane, on the other hand, will not be thus discolored, but will be badly infested with bacteria and moulds. When the sugar cane is attacked by the borer or beetle, the pathway of the insect is much discolored, but we notice no inroad of organisms into the sound tissues. The living plant therefore does appear to protect itself against the invasion of microscopic parasites by forming toxic products. In case the sugar cane is killed, as sometimes happens during a freeze, this power of protection is lost. The formation of toxic products does not go on, hordes of bacteria invade the cane, and finding no resistance, start a fermentation which soon renders the cane worthless for milling.

The question may be asked if these toxic products are so deadly to micro-organic life, why do they not react unfavorably upon the cane itself? It is just here that the reducing or catalyzing enzymes perform their functions, for should the toxic oxidation products diffuse inward beyond the points of their formation, they are at once reduced and thus exert no action deeper than the exposed surface.

It will be seen that there must be a great difference between working with enzymes before and after expression from the cane. As Pfeffer remarks in his Plant Physiology, "The living cell must not be judged by reactions obtained with dead material or in the expressed juice." That there is a localization of the enzymes within the plant is rendered very evident if we apply the guaiac test to the cross section of a cane stalk; the blue coloration is developed the strongest upon the peripheral parts, showing that the oxidases are apparently more localized in this region. Pozzi Escot* has demonstrated that the same condition exists

*American Chemical Journal, June 1903, page 530.

with other plants such as the potato; his experiments show, however, that the oxidases exist in the inner tissues as well, but that the greater localization of the reductases in these parts interferes with the guaiac reaction. Jacoby* goes even a step further and states that the localization of ferments exists not only in the tissues, but within the individual cell. He sums up the situation very briefly in these words: "The work of the cell can be determined with absolute certainty only when we learn to separate in the test tube the substances which are separated in the cell, and allow the individual ferments to do their work in proper sequence and in proper concentration."

In addition to the enzymes named there are other natural ferments which play an important part in the vital processes of the cane. In the leaf we have evidences of the activity of a diastase by which the starch that is formed in the chlorophyll grains is converted into sugar. We have already called attention to the presence in the mother cane of peptonizing enzymes, by which the albuminoids are broken down into soluble nitrogenous compounds easily transportable to the young plant.

It will be impossible to dwell longer upon the functions and properties of these various ferments which occur within the cane, enough having been said, however, to indicate the physiological importance of this class of compounds.

IV. CONDITIONS AFFECTING THE COMPOSITION OF THE SUGAR CANE.

There are a number of factors having an influence upon the composition of the sugar cane that require a brief consideration. Among these may be mentioned the influence of climatic conditions, influence of variety of cane, influence of cultivation, and influence of soil and fertilization.

INFLUENCE OF CLIMATIC CONDITIONS UPON COMPOSITION OF CANE.

Reference has been made to the influence of climatic conditions upon the ripening of cane. This can best be seen by comparing analyses of juices at different periods for the successive years 1903 and 1904, which were very dissimilar as regards weather conditions.

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*Ergebnisse der Physologie I, 1, page 213.

TABLE VIII.

1903.

	Aug. 1.	Sept. 1.	Oct. 1.	Nov. 1.	Nov. 15.
Sucrose	2.70	5.97	11.27	13.60	15.86
Glucose	3.80	3.68	2.51	1.02	.63
Purity	36.00	57.02	76.72	87.85	92.10

1904.

	Aug. 1.	Sept. 1.	Oct. 1.	Nov. 1.	Nov. 15.
Sucrose	2.35	5.13	8.04	9.13	12.00
Glucose	4.04	3.75	3.55	2.82	1.66
Purity	32.28	52.35	66.61	71.55	80.53

The weather conditions for the past two years in question during the growing season were as follows:

	July.	Aug.	Sept.	Oct.	Nov.	Dec.
Av. daily temperature, F., 1903	82.80	82.10	77.00	67.90	58.00	49.00
Av. daily temperature, F., 1904	80.90	84.10	84.20	71.20	64.30	58.40
Rainfall inches, 1903	5.55	5.98	1.27	0.36	0.23	3.89
Rainfall inches, 1904	6.47	5.75	3.24	0.75	1.50	3.02

The results for the two years show but little variation up to the middle of September. After this date the canes of 1903 increased considerably faster in sucrose, and this increase continued until the end of the season, when it exceeded 3 per cent. The average daily temperature and rainfall for the two years were also about the same during June, July and August; for the remaining months of the year, however, the conditions were very unlike. September, October, November and December of 1903 showed daily averages in temperature of 7, 3.3, 6.3 and 9.4 degrees respectively lower than the corresponding months of 1904; 1903 also showed a deficiency of rainfall of 1.97, 0.39 and 1.27 inches for the months of September, October and November. These conditions for 1903 were very adverse to the growth of cane, yet hastened the ripening to an extent rarely attained in Louisiana. On the other hand, the unusually warm weather

of the fall of 1904, together with favoring rains, promoted the growth of canes even into December, but retarding the ripening. The tonnage was high, but the sucrose content low. These differences in conditions naturally made themselves very noticeable in the sugar house, owing to the much lower purities of the juices and the relatively larger amount of water requiring evaporation.

INFLUENCE OF VARIETY UPON THE COMPOSITION OF CANE.

That the different varieties of the sugar cane differ from one another in the sugar content and purity of their juices is too well known to require special mention. These differences, however, are not confined to sugar content, but extend to the other ingredients of the cane as well. The physiological peculiarities of the different canes are as well marked as the differences in color or shape, and are much more potent as regards effect upon the chemical composition.

An interesting fact in the above connection is the matter of ash content. We have in the ash an imperfect yet a fairly comparative measure of the transpired water for each variety of cane, since the mineral matter in solution as it enters the plant from the soil, accumulates in proportion to the degree of evaporation from the leaf surface. In the following table are given a few results taken from analyses of juices made the middle of August, which is about the period of most rapid growth:

TABLE IX.

	D. 74.	D. 95.	Purple.	Striped.
Sucrose	4.88	2.45	2.35	2.03
Glucose	3.24	3.87	4.04	4.26
Ash	.48	.41	.40	.34

The ash content is much higher in the juice of D.74 than in any of the other varieties; D.95 stands second, Purple third and Striped fourth. This is also their relative position in sucrose content; as regards reducing sugars the order is the reverse of this. From the above results, which hold true during the entire period of growth, we may conclude that of the several canes studied, the D.74 is the most vigorous feeder, thus requiring

upon poor soils a heavier fertilization; we may also say that the D. 74 has the greatest powers of assimilation and conversion, as is shown by the higher content in sugars and lower glucose ratio. The experiments upon the different varieties are to be continued during the present season and a more complete synopsis of the results will be presented later.

The effect of variety upon the composition of the ash of the juice will be shown in a subsequent table.

INFLUENCE OF CONDITIONS OF CULTIVATION UPON COMPOSITION OF CANE.

Conditions of cultivation have a very marked influence upon the composition of sugar cane. Good tilth and thorough cultivation favor the growth of the cane, whereas a poor condition of the soil has the opposite effect. The difference in growth and composition can best be appreciated by comparing the analyses of plant and stubble canes. The following table gives the composition of Striped and D.74 canes for *plant, first year stubble* and *second year stubble,* during the latter part of September, 1904:

TABLE X.
(Analyses by Chiquelin and Verret.)

		Plant.	1st year Stubble.	2d year Stubble.
Striped cane.	Weight stalk	1894 gm.	1262 gm.	1042 gm.
	Fiber	6.56 %	7.45 %	8.02 %
	Sucrose	4.79	6.03	8.45
	Dextrose	2.05	2.27	1.97
	Levulose	1.60	1.73	1.64
	Ash	0.39	0 27	0.27
	Acids	0.21	0.18	0.11
	Albuminoids	0.08	0.06	0.07
	Amids	0.08	0.02	0.02
	Gums	0.06	0.07	0.08
D. 74 cane.	Weight stalk	1575 gm.	1497 gm.	1163 gm.
	Fiber	6.28 %	7.12 %	7.16 %
	Sucrose	6.33	7.36	8.24
	Dextrose	1.84	1.65	1.83
	Levulose	1.35	1.20	1.12
	Ash	.40	.41	.39
	Acids	.21	.22	.18
	Albuminoids	.06	.04	.07
	Amids	.10	.03	.02
	Gums	.07	.07	09

It will be noted that the stubble canes average considerably less in weight than the plant canes. A comparison of various experiments made at Audubon Park shows that first year stubble averages about 80 per cent, and second year stubble about 70 per cent of the weight of plant cane. The stubble canes on the other hand are richer in fiber and sucrose than the plant, the second year stubble exceeding the first in these respects.

There is, of course, a physiological explanation for these differences. In stubble cane we have a partially dwarfed condition and according to a well established law, when growth is checked, maturation is hastened. Exactly the same effect is produced by the non-fertilization of cane. Canes grown upon the non-manured plots at the Sugar Experiment Station average much less in weight, but are higher in sucrose than canes which have been fertilized. The stunted growth of our stubble cane is due very largely to the inability of the crop to secure a sufficient supply of plant food, particularly nitrogen; an indication of this is shown by the deficiency of the juices from stubble cane in mineral and in nitrogenous ingredients. An inspection of the table will show this very clearly.

A comparison of many analyses made at Audubon Park upon the leading varieties of cane shows that stubble cane contains from 10 per cent to 20 per cent less ash and over 50 per cent less nitrogen than plant canes. The analyses also show that the deficiency of nitrogenous ingredients in stubble canes falls most largely upon the reserve supply of nitrogen or the amids. This is due to the greater nitrogen hunger of the stubble canes.

To maintain the vital processes of the cane a certain amount of albuminoid matter (protoplasm) is indispensable and to keep this up the plant draws upon its store of amids. An inspection of the table shows that with the stubble canes this reserve is almost completely exhausted and subsequent analyses showed no gain. With the plant canes, on the other hand, there is always a large surplus of amids, which is being continually added to during the entire period of growth.

There are a number of reasons for this partially starved condition of our stubble crops. Among the most important of these may be mentioned the partial exhaustion of fertility directly beneath the roots, by the previous crop, the difficulties in

securing good tillage and the very unfavorable points of growth. The most that can be done towards helping the situation is to cultivate as thoroughly as possible and to fertilize well with nitrogenous manures.

INFLUENCE OF SOIL AND FERTILIZATION UPON COMPOSITION OF CANE.

Differences in the composition of soil naturally influence to a greater or less extent the composition of the cane and its products. This fact is well known to every one who has compared the taste of syrup or molasses from the salty cane fields of the lower coast with that from plantations which have not been subject to floodings of salt water. Canes from the lower coast contain sometimes four times the amount of chlorides as canes raised above New Orleans. The presence of an excess of salt in the soil affects very seriously the growth of the cane and also introduces various irregularities into the distribution of the different constituents.

Reference was previously made to the marked effect of fertilization and non-fertilization upon the composition of sugar cane. The effects of different fertilizers upon the composition of the cane and its juices is also well marked, but this subject would require so much space to be adequately treated that it has been reserved for a separate bulletin.

V. COMPOSITION OF SUGAR-CANE JUICE AND METHODS OF EXTRACTION.

Theoretically the juice of the cane is simply a solution of the soluble ingredients, sugars, salts, acids, etc., in the cell-water. The juice as expressed from the mill, however, contains in addition to the soluble matter a small amount of the insoluble ingredients of the cane, such as particles of fiber, wax and fat, albuminoids, dirt, etc., in suspension or in emulsion so that a filtration of the juice without previous clarification is almost an impossibility.

The composition of the average juice in Louisiana as it is obtained undiluted from the mill is given in Table XI.

TABLE XI.
(Composition of Cane Juice.)

Water	85.00	per cent.	
Ash	0.40	"	
Nitrogenous bodies	0.26	"	
Sucrose	12.00	"	
Dextrose	1.00	"	
Levulose	0.70	"	
Free Acids	0.10	"	
Combined Acids	0.15	"	
Pectin	0.10	"	
Fat and wax	0.10	"	Mechanical impurities
Fiber particles	0.12	"	removed from the cane
Dirt and earthly matter	0.06	"	during milling.
Tannin, coloring matter, etc.	0.01	"	
	100.00		

The composition of the ash from the juice of different varieties of cane according to analyses by Hall is given in Table XII.

TABLE XII.
(Composition of Ash from Sugar Cane Juice.)

	Home Canes.		Seedling Canes.	
	Purple.	Ribbon.	Demerara 74	Demerara 95
Potash K_2O	49.63 %	47.65 %	44.21%	40.66 %
Soda Na_2O	1.81	1.13	1.11	0.63
Lime CaO	3.00	3.79	4.62	4.33
Magnesia MgO	3.21	3.73	7.45	5.70
Iron Oxide Fe_2O_3	0.70	0.45	1.15	1.48
Alumina Al_2O_3	0.40	0.32	0.18	1.25
Silica SiO_2	4.80	5.92	6.20	9.30
Phosphoric Acid P_2O_5	5.80	6.38	5.31	5.39
Sulphuric Acid SO_3	20.40	20.59	22.46	23.69
Carbonic Acid CO_2	4.10	3.30	3.40	3.40
Chlorine Cl	5.80	5.83	5.36	3.26
Total	99.65	99.09	101.45	99.09
Deduct O=Cl	1.31	1.32	1.21	0.74
	98.34	97.77	100.24	98.35
Carbon and undetermined	1.66	2.23		1.65
Alkalinity ($cc_{\frac{n}{10}}$ per gm. ash)	36 cc	30 cc	24 cc	22 cc

The canes were all taken from the same plot and were grown under perfectly similar conditions of cultivation and fertilization. While all the analyses show a certain uniformity,

the difference in composition between the ash of the home canes and the seedlings is well marked.

The distribution of the nitrogen among the different ingredients of cane juice is shown from the following analyses by Hardin in Table XIII.

TABLE XIII.
(Distribution of Nitrogen in Cane Juice.)

	Percentage in Juice.	Percentage of Total Nitrogen.
Nitrogen in albumen	.0039%	9.47%
" " nuclein bodies	.0025	6.32
" " albuminoses	.0021	5.26
" " amido acids,(Aspartic)	.0122	30.53
" " amido acid amids (Asparagin)	.0098	24.07
" " ammonia	.0024	6.18
" " nitrates	.0071	17.77
" " nitrogenous bases		
Total	0.0400	100.00

VARIATIONS IN THE COMPOSITION OF SUGAR CANE JUICE.

The various factors previously enumerated, climate, variety, cultivation, fertilization, etc., which affect the composition of the cane as a whole, also naturally affect the composition of juice, and require no further consideration at this point. There are several factors, however, that have not yet been touched upon, which have an influence upon the composition of the juice and which should be discussed before taking up the subject of clarification.

VARIATIONS IN COMPOSITION OF CANE JUICE DUE TO MANNER OF CUTTING, ETC.

As was previously mentioned, the top of the cane contains less sucrose and more of the solids not sugar than the lower portions of the stalk. The manner of topping canes, whether low or high, will therefore have considerable effect upon the composition of the juice. The following analyses by Messrs. Agee and Hall at Audubon Park, giving the composition of the juice from different parts of the cane for several varieties, illustrate this very clearly.

TABLE XIV.

(Composition of juice from different joints of the sugar cane.)

TABLE XIV.
(Composition of Juice from different joints of the Sugar Cane.)

No. of Joint	Purple				Striped				D. 74				D. 95			
	Wt. Joint in grams	Brix	Sucrose	Glucose	Wt. Joint in grams	Brix	Sucrose	Glucose	Wt. Joint in grams	Brix	Sucrose	Glucose	Wt. Joint in grams	Brix	Sucrose	Glucose
1..... Butt	66.0	13.2	10.20	1.25	112.0	14.7	12.35	0.96	155.5	16.8	14.80	0.42	106.0	15.9	14.40	0.25
2.....	86.5	13.1	10.30	1.53	128.5	14.6	12.15	1.04	170.5	16.9	15.00	0.31	139.0	15.9	14.40	0.29
3.....	99.5	13.0	10.10	1.64	130.5	14.6	12.00	1.22	173.5	16.8	15.00	0.31	154.5	15.9	14.30	0.33
4.....	101.5	13.2	10.50	1.72	135.5	14.4	11.75	1.31	183 5	16.7	14.75	0.31	145.5	16.0	14.40	0.36
5.....	101.0	13.1	10.10	1.72	132.0	14.2	11.50	1.42	181.5	16.7	14.70	0.35	140.5	16.3	14.60	0.35
6.....	100.0	13.2	10.25	1.72	129.0	14.1	11.15	1.51	172.0	16.6	14.50	0.39	120.0	16.3	14.70	0.38
7.....	90.0	12.9	9.85	1.85	119.0	14.3	11.10	1.66	159.0	16.5	14.25	0.44	103.0	16.6	14.90	0.35
8.....	64.0	13.0	9.70	2.08	106.0	14.1	11.10	1.66	148.9	16.5	13.80	0.59	96.0	16.7	14.90	0.38
9.....	60.0	12.7	9.00	1.88	82.0	14.0	10.75	1.66	139.0	16.4	13.50	0.78	92.0	16.6	14.70	0.46
10.....	55.0	12.6	8.90	1.92	71.0	14.0	10.50	1.72	127.5	16.5	13.30	0.74	84.5	16.7	14.60	0.49
11.....	48.5	12.4	8.55	2.04	64.5	14.0	10.40	1.85	111.5	16.4	13.20	0.83	81.5	16.3	14.00	0.74
12.....	44.5	12.3	8.00	2.04	57.5	13.6	9.70	1.88	101.5	16.5	13.10	1.00	81.5	16.0	13.60	0.85
13.....	43.0	12.0	7.20	2.12	54.5	13 4	9.40	1.92	89.0	16.6	12.90	1.13	75.5	16.2	13.50	1.04
14.....	42.5	11.0	6.60	2.17	48.0	12.4	8.35	2.08	54.5	15.3	11.70	1.28	57.5	15.4	11.90	1.61
15.....	34.5	10.5	5.50	2.27	36.0	11.4	7.30	2.22	29.5	13.8	8.60	1.88	37.0	14.0	9.10	2.38
16..... Top	22.2	9.8	5.40	1.53	32.0	10.0	5.50	2.00	24.0	10.9	4.20	2.12	30.8	11.6	5.50	3.03

The above analyses represent each the averages of ten stalks of plant cane, cut and topped according to the methods usually followed in Louisiana. The solids not sugar in the juice increase as we go up the stalk, taking a sudden jump in the last two or three joints. In some cases the purity of the juice from the upper joints is less than one-half that from the butt of the cane. The extreme top of the cane as it is cut in Louisiana, has but little value for sugar making, as can be seen from the foregoing analyses. If the cane were topped several joints lower and the top reserved for seed as in tropical countries, juices of higher sucrose content and purity would be obtained and a great saving effected in the amount of cane reserved each year for planting.

The difference in composition of the juice from the nodes and internodes of the sugar cane has been studied by Beeson and the following results are quoted from his paper in Bulletin 38, Louisiana Sugar Experiment Station.

TABLE XV.

(Composition of Juices from Nodes and Internodes. Beeson,)

		Total solids.	Reducing sugars.	Sucrose.	Solids not sugar.
Tops	Nodes	15.5	0.66	12.7	2.64
	Internodes	16.8	1.20	15.0	1.60
Middles	Nodes	15.2	0.20	13.5	2.90
	Internodes	17.6	1.00	15.6	1.00
Butts	Nodes	14.2	0.26	11.9	2.04
	Internodes	17.2	0.89	15.1	1.21

The difference in composition of the juice within the cane is not only noticeable in the different joints, and in the nodes and internodes, but it is also very marked between the different tissues, the pith and the fibro-vascular threads or bundles. The juice from the latter is frequently forced out from the end of the cane when it passes through the rollers of the mill and analyses show it to be almost completely deficient in sugar. This phenomenon has been very clearly explained by Dodson, who has shown by numerous experiments that the juice thus expressed from the ends of the cane is simply a solution of

mineral matter from the soil on its way through the vascular tubes to the leaf. "Though it traverses very near the tissues rich in sugar, it contains but little organic matter of any kind."

VARIATIONS IN COMPOSITION OF JUICE DUE TO METHODS OF EXTRACTION.

In the extraction of juice from sugar cane by the mill, the amount of mechanical impurities introduced into the juice will vary according to the pressure of the rollers. Accordingly we would expect juice of a higher purity from the first mill or crusher than is the case with juice from the last mill. That this is exactly the result of practical experience may be seen from the following series of experiments made at Audubon Park in November, 1903:

TABLE XVI.

Purple Cane—Second Year Stubble.

Extraction first mill	64.50%
" second mill	5.50
" third mill	2.13
Total extraction	72.13%

	First Mill.	Second Mill.	Third Mill.
Brix	15.36	14.60	14.60
Sucrose	12.93	11.41	11.30
Glucose	1.54	1.29	1.23
Ash	0.37	0.58	0.77
Albuminoids	0.18	0.50	0.58
Free acid	0.10	0.11	0.14
Combined acid	0.14	0.15	0.12
Gums	0.10	0.56	0.51
Coefficient of purity	84.07	78.15	77.29
Glucose ratio	11.91	11.30	10.88

The great increase in the ash, albuminoids and gums in the juice of the third mill is especially noteworthy, the coefficient of purity showing a decrease of nearly seven units from that of the first mill. If water or steam be employed for saturation in connection with the milling the percentages of impurities in the juice will be still further increased as can be seen from the following two series of experiments carried out at the same time as the previous one:

TABLE XVII.

Cold water saturation (20 per cent)—Purple Cane-plant.

Extraction first mill	66.50%
" second mill	12.15
" third mill	4.51
Total extraction	76.83%

	First Mill.	Second Mill.	Third Mill.
Brix	15.73	10.48	6.93
Sucrose	14.01	8.65	5.64
Glucose	0.83	0.46	0.34
Ash	0.35	0.37	0.31
Albuminoids	0.12	0.22	0.21
Free acid	0.08	0.07	0.06
Combined acid	0.10	0.09	0.08
Gums	0.24	0.62	0.29
Coefficient of purity	89.07	82.53	81.38
Glucose ratio	5.92	5.32	6.03

TABLE XVIII.

Steam saturation—Purple cane—first year's stubble.

Extraction first mill	64.31%
" second mill	8.00
" third mill	4.52
Total extraction	76.83

	First Mill.	Second Mill.	Third Mill.
Brix	15 84	12.05	11.53
Sucrose	13.50	9.40	8.70
Glucose	1.37	0.97	0.88
Ash	0.37	0.57	0.65
Albuminoids	0.14	0.28	0.20
Free acid	0.09	0.13	0.14
Combined acid	0.13	0.16	0.19
Gums	0.24	0.54	0.83
Coefficient of purity	85.23	78.00	75.45
Glucose ratio	10.01	10.32	9.42

In the experiment with steam saturation a jet of steam is introduced through the turn plates between the rollers of the second and third mills. The hot steam effected a coagulation of the albuminoids, so that less of these were removed than in the process of dry extraction; on the other hand, the percentage of gums removed from the cane was very much increased with the result that the juice obtained by steam saturation was very difficult to clarify.

A considerable difference is noticeable in the composition of juices as obtained by the mill and by the diffusion battery. The following comparative experiments made by Beeson at Audubon Park, will illustrate this very clearly. The diffusion juice owing to its dilution was calculated to the same content of solid matter as the mill juice:

TABLE XIX.

Juice.	Total solids	Suc.	Gluc.	Solids not sugar.	Coeff. purity.	Gluc. ratio.	Ash.	Albuminoids.	Amids.	Gums, acids, etc.
Mill	14.9	11.9	1.40	1.60	80.0	11.8	0.41	0.071	0.060	0.96
Diffusion	14.9	12.14	1.57	1.29	81.5	12.9	0.48	0.044	0.071	0.70

It will be noted that the percentage of mechanical impurities such as gums, etc., is much higher in the mill juices. The hot water used in diffusion caused, on the one hand, a coagulation of a large amount of albuminoid matter, which was held back in the diffusion chips, but on the other effected the solution of a larger amount of amids and mineral matter. Maxwell has also shown (Bulletin 38, Louisiana Sugar Experiment Station) that "diffusion may take out, particularly at high temperatures, more of some bodies and less of others than are found in the normal mill juice."

VI. CLARIFICATION OF SUGAR CANE JUICE

Cane juice, as has been seen, contains in addition to sucrose, varying amounts of other ingredients, organic and inorganic, the removal of which constitutes the first and in many respects the most important operation of the sugar house. In this process of clarification, as it is called, certain of the impurities of the juice, especially those of a mechanical nature, such as fat

and wax, fiber, soil, etc., may be eliminated entirely. Other impurities such as the ash, albuminoids, acids, and gums are even in the best of our present processes only partially removed, while other undesirable ingredients such as the reducing sugars, and amids are not at all precipitated by the ordinary clarifying agents employed. The work of clarification affects the composition of the cane juice by the removal of certain ingredients either in whole or in part; also by the transformation of certain of these constituents into other forms—the sucrose for example may be partially inverted, the glucose changed into acid products, or the amids and albuminoids converted into substances of a different character. The chemistry of the various processes of clarification is therefore exceedingly complex and the character of the changes which take place is in many instances very hard to follow owing to our imperfect knowledge of many of the products formed.

In our present study of the processes of clarification we will take up the action of heat alone upon juices and then the action of lime, both alone and in conjunction with sulphur dioxide. Phosphoric acid and carbonic acid, certain special processes such as superheating and electric clarification, will also be briefly discussed.

ACTION OF HEAT ALONE IN THE CLARIFICATION OF CANE JUICES.

Heat alone for clarification is never employed in the sugar house, owing, first, to the imperfect removal of many of the impurities from the juice and more especially to the great loss of sucrose from inversion by the organic acids. In the manufacture of cane syrup, however, heat is often the only means of clarification employed, since a partial inversion of sucrose for this purpose may be even desirable to prevent crystallization.

The following experiments by Agee and Hall upon clarification by heat were performed on juices of the Striped and D.74 canes. The juice after weighing was boiled for periods of one and two hours. After cooling the juice was reweighed and sufficient water added to make up for the loss by evaporation. The juices were then filtered and analyzed.

TABLE XX.

Striped Cane.

	Raw juice.	Boiled one hour.	Boiled two hours.
Total Solids	13.82	13.59	13.59
Sucrose	11.26	11.12	10.97
Reducing Sugars	1.34	1.47	1.61
Ash	0.43	.....	.0.44
Albuminoids	0.061	.....	0.02
Amids	0.176	.....	0.165
Free Acid as Malic	0.11	0.10	0.10
Gums, etc.	0.44	.....	0.28
Coefficient of Purity	81.47	81.82	80.72
Glucose Ratio	11.90	13.22	14.68

D.74 Cane.

	Raw juice.	Boiled one hour.	Boiled two hours.
Total Solids	15.93	15.48	15.48
Sucrose	14.47	14.27	14.14
Reducing Sugars	0.34	0.51	0.67
Ash	0.49	.....	0.45
Albuminoids	0.078	.....	0.014
Amids	0.081	.....	0.085
Free Acid as Malic	0.08	0.07	0.07
Gums, etc.	0.39	.....	0.05
Coefficient of Purity	90.83	92.18	91.34
Glucose Ratio	2.42	3.57	4.74

The experiments show that simple boiling removed 0.23 per cent of impurities in the juice from the Striped cane and 0.45 per cent from that of the D.74. The impurities removed consisted largely of gums and albuminoids with a small amount of acids and ash. The glucose ratio shows a steady increase throughout the experiment, showing a marked inversion of sucrose. The purities show a decided increase at first, owing to the rapid coagulation of the albuminoids and gums and then a gradual falling off as the process of inversion continues.

In discussing the various clarifications by chemical means, the numerous processes have for convenience of description been

divided into two general classes, *Alkaline Clarification*, where the juice is first treated with milk of lime, and *Acid Clarification*, where the juice is first acidified with sulphurous or phosphoric acid. In a properly regulated clarification the juice is of course brought back to neutrality before boiling, so that the above classification refers only to the preliminary treatment of the juice and not to its condition after clarification.

Alkaline Clarifications.

ALKALINE CLARIFICATION WITH LIME ALONE.

Lime has been used from the very earliest times for the clarification of cane juices and for cheapness, availability, and general excellence no other clarifying agent has been found to take its place. The first action of lime when added to cane juice consists in neutralizing the organic acids. If heat is then applied no inversion of sucrose takes place; the albumin of the juice is coagulated, the sulphates and phosphates are thrown down as insoluble lime salts, and the bases, iron and alumina, precipitated. The separation of these impurities also exerts a mechanical purification, the fat and wax, fiber, particles of soil, and a portion of the gums being removed by the formation of the flocculent precipitate. The precipitate produced by lime and heat collects into two portions—the one containing a greater part of the lighter bodies, such as wax and fiber, rising to the surface where it forms the blanket or scum, and the other part, comprising more of the heavier substances, dirt, lime, sulphate, etc., forms the settlings.

The approximate composition of the scums and settlings can be seen from the following analyses by Beeson:

	Ash.	Albuminoids.	Fat, wax, fiber, acids, gums, etc.
Scums	21.9	18.9	59.2
Settlings	23.7	15.6	60.7

An excess of lime in clarification is to be avoided. Any amount beyond what is required for the neutralization of the acids and the precipitation of impurities exerts a destructive action upon other constituents of the juice. The reducing sugars are especially attacked being converted into soluble lime compounds such as the glucinate of lime, the dark color of which injures the appearance of the syrup and final products. These lime salts also increase the viscosity of the juice thus retarding the work of evaporation. The removal of lime from these salts by mineral acids such as phosphoric has been proposed, but this does not result in the reformation of glucose. Instead the glucinic acid is liberated and remains in solution, retaining its dark color and other injurious properties. An excess of lime also acts upon certain of the amids converting the asparagin into aspartic acid and ammonia, which escaping into the air accounts for the smell of ammonia occasionally noticeable around clarifiers. The excess of lime may also act upon some of the precipitated albuminoids giving rise to various amido and fatty acids with the liberation of some ammonia. The glucinate of lime has itself an action similar to that of free lime, so that clarified juices weakly alkaline to begin with may in the syrup show a slight acidity owing to the formation of acid products during boiling.

A large number of experiments showing the action of heat and different amounts of lime upon cane juice were carried out at Audubon Park in 1898. The following examples showing the effects of an acid, a neutral and an alkaline clarification with lime; are cited. The samples for analysis were taken from the same clarifier before and after clarification. All results were calculated to the same sucrose content for comparison.

Experiment I.

Juice had an acidity of 1.6 cubic centimeters, and lime added till .55 cubic centimeter acidity remained, requiring .0384 ounce of lime per gallon.

TABLE XXI.

MILL JUICE.

Acidity in cubic centimeters	1.600
Brix	14.610
Sucrose	12.000
Glucose	1.140
Solids not sugar	1.470
Purity	82.130
Glucose ratio	9.500
Proteids	.179
Albuminoids	.083
Amids	.096
Alcoholic precipitate	.181
Gums, etc	.074
Ash in alcoholic precipitate	25.300
Albuminoids in alcoholic precipitate	35.430

CLARIFIED JUICE.

Acidity in cubic centimeters	.550
Brix	14.460
Sucrose	12.000
Glucose	1.140
Solids not sugar	1.320
Purity	82.980
Glucose ratio	9.500
Proteids	.128
Albuminoids	.029
Amids	.099
Alcoholic precipitate	.107
Gums, etc	.048
Ash in alcoholic precipitate	46.870
Albuminoids in alcoholic precipitate	8.190

An increase in purity of .85 was caused by the removal of some of the solids not sugar. The sucrose and glucose were not affected. Of the impurities 28.4 per cent of the proteids, consisting entirely of albuminoids, were removed, the amids remained unchanged and 35 per cent of the gums were removed.

Experiment II.

Neutral clarification, mill juice with an acidity of 1.8 cubic centimeters, lime added to neutrality, required .068 ounce of lime to one gallon of juice.

TABLE XXII.

MILL JUICE.

Acidity	1.800
Brix	14.850
Sucrose	12.000
Glucose	1.460
Solids not sugar	1.390
Purity	80.800
Glucose ratio	12.170
Proteids	.138
Albuminoids	.069
Amids	.069
Alcoholic precipitate	.212
Gums, etc	.071
Ash in alcoholic precipitate	33.540
Albuminoids in alcoholic precipitate	34.350

CLARIFIED JUICE.

Acidity	.000
Brix	14.610
Sucrose	12.000
Glucose	1.450
Solids not sugar	1.150
Purity	82.130
Glucose ratio	12.090
Proteids	.086
Albuminoids	.013
Amids	073
Alcoholic precipitate	.099
Gums, etc	.058
Ash in alcoholic precipitate	29.090
Albuminoids in alcoholic precipitate	12.750

An increase in purity of 1.33 is noted. The glucose is not affected, but there is a decrease in the brix and solids not sugar; 38 per cent of the proteids are removed consisting entirely of albuminoids, the amids remaining practically the same; 53 per cent of the alcoholic precipitate and 18 per cent of the gums are also removed.

Experiment V.

Juice limed to 3.3 cubic centimeters alkalinity, the acidity before liming being 1.9 cubic centimeters, .1953 ounce of lime added per gallon of juice.

TABLE XXIII.

MILL JUICE.

Acidity	1.900
Alkalinity	———
Brix	14.280
Sucrose	12.000
Glucose	1.540
Solids not sugar	.740
Purity	84.030
Glucose ratio	12.530
Proteids	.133
Albuminoids	.056
Amids	.067
Alcoholic precipitate	.172
Gums, etc	.046
Ash in alcoholic precipitate	36.820
Albuminoids in alcoholic precipitate	39.000

CLARIFIED JUICE.

Acidity	———
Alkalinity	3.300
Brix	14.370
Sucrose	12.000
Glucose	1.210
Solids not sugar	1.160
Purity	83.510
Glucose ratio	10.080
Proteids	.084
Albuminoids	.017
Amids	.067
Alcoholic precipitate	.212
Gums, etc	.107
Ash in alcoholic precipitate	46.340
Albuminoids in alcoholic precipitate	3.120

There is a decrease of .52 in the purity and an increase of .42 in the solids not sugar, a destruction of 21.4 per cent of the glucose and a corresponding decrease in the glucose ratio. The increase of 56.8 per cent in the solids not sugar, notwithstanding the removal of 37 per cent of proteids, shows the deleterious results of excessive liming.

The percentage of amids remains the same, but this does not indicate that none of them were converted to organic acids, as strong alkalinity decomposes the albuminoids into amids and ammonia, and the amount of decomposed albuminoids in this instance formed enough amids to equal those decomposed by the lime. The gums increase 137 per cent, while the alcoholic precipitate increases 23 per cent. The large increase in the gums is due to the organic acids formed by the action of the excess of lime, and the comparatively small increase in the alcoholic precipitate is due largely to the removal of the albuminoids from the juice; the albuminoids in the alcoholic precipitate of the mill juice were 39 per cent, while those in the alcoholic precipitate of the clarified juice were only 3.12 per cent.

These experiments show conclusively that an excess of lime decomposes the glucose and amids to a considerable extent and forms acids of a gummy character, and when the lime is largely in excess, the amount of these acids formed is sufficient to increase the gums over 100 per cent, thus more than doubling the amount of the most objectionable impurities in the juice, and at the same time darkening it by their presence. Besides this, which of itself is sufficient to condemn liming to alkalinity, the excess of lime acting upon the albuminoids (which are largely removed by heat alone, though aided by the addition of lime) converts them into amids, which remain in the juice, and further acting on these amids, decomposes them into organic acids and ammonia. The results of both these actions bring back into solution impurities that have been removed and are decidedly disastrous in sugar manufacture.

By leaving the juice slightly acid there was no addition of impurities, but the amount removed was not as great as that in the neutral clarification. The gums, of which a larger quantity is removed in the acid clarification given here than in the

neutral clarification, were undoubtedly those obtained from the cane by light pressure as in the neutral clarification, lime not acting on these as strongly as on those removed by increasing the pressure.

The best method to clarify with lime alone is to add lime either to neutrality or slight acidity.

The method of clarification has necessarily a great deal to do with the composition of the after-products. An experiment made at Audubon Park in 1904, where lime only was used for clarification, showed the results tabulated in Table XXIV.

The work confirms the results previously reported. Ten per cent of the mineral matter of the juice is removed. The clarified juice and syrup show a marked acidity, notwithstanding that the raw juice was previously neutralized; this is undoubtedly due to the formation of acid decomposition products of the albuminoids, a supposition borne out by the increase of the amids. This decomposition of albuminoids and increase in amids goes on continuously through the whole process of evaporation and boiling, the ratio of albuminoids to amids being one to three in the syrup, one to four in the first molasses and one to five in the second molasses. The formation of amids, etc., is even more marked than is indicated by this increase in ratio for there is a considerable loss of nitrogen especially in the last stage of the process. In a number of experiments from both alkaline and acid clarification it was found that in boiling the second molasses to masse cuite there was a loss of 15 per cent of the total nitrogen. It was not determined in just what form this nitrogen escaped. It may have been in the form of ammonia or perhaps even in the form of gaseous nitrogen.

ALKALINE CLARIFICATION WITH LIME AND PHOSPHORIC ACID.

In the following experiment performed at Audubon Park in 1904, the juice was treated with a slight excess of lime and then after heating and skimming the greater part of the excess of lime was removed with phosphoric acid:

TABLE XXIV.

Juice 1.2° C. C. Acid.

Limed to Neutrality. Mixed Varieties of Cane.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	14.55	11.47	0.98	1.08	78.83	17.96	0.40	0.15	0.12	0.15	0.097	0.085
Clarified Juice	14.63	11.54	0.97	1.03	78.88	17.33	0.36	0.06	0.15	0.17	0.034	0.104
Syrup	50.00	39.29	3.64	3.41	78.59	17.94	1.37	0.11	0.55	0.42	0.145	0 350
First Masse Cuite	90.00	74.05	6.17	6.22	82 28	16.74	2.34				0.218	0.611
First Sugar		93.60	3.14									
First Molasses	80.00	46.07	11.87	11.98	57.58	51.80	4.90				0.362	1.275
Second Masse Cuite	90.00	51.64	12.75	14.05	57.37	51 90	5.40				0.376	1.446
Second Sugar		88.45	4.26									
Second Molasses	80.00	31.26	15.46	17.04	39.07	104.0	7.04				0.399	1.893
Third Masse Cuite	90.00	33.26	15.71	17.91	36.96	101.1					2.152	

The excess of lime resulted in a destruction of over 15 per cent reducing sugars with a corresponding increase in the percentage of gums and combined acids. The action of lime seems to have continued as long as the products of the juice were worked in the sugar house, as it will be noted that there is a marked falling off in the glucose ratio with each boiling.

The concentration of mineral matter in the final products resulted in the precipitation of much calcium phosphate, causing a marked turbidity in the final molasses, and giving rise to a large amount of insoluble mineral matter in the third sugars (not reported in the table).

The excess of lime resulted in the destruction of a larger amount of albuminoids than in the preceding experiment with a corresponding increase in the albuminoid-amid ratio between the syrup and final molasses.

A striking fact in connection with this and the other clarification experiments, is the change which takes place in the ratio of dextrose to levulose during the process of manufacture. The dextrose which was in excess in the raw juice is practically equal to the levulose percentage in the first molasses, but in the second masse cuite resulting from this same molasses the dextrose is much lower than the levulose and this difference is even more pronounced in the final molasses.

It must of course be remarked that our methods of estimating dextrose and levulose in such a complex medium as molasses fall considerably short of perfection; nevertheless the results plainly indicate a greater preponderance of levorotatory substances in the last sugar house products, showing either a formation of new left-rotating substances, or a transformation of a part of the dextrose into bodies which polarize less to the right. The latter alternative is extremely probable for it is a well known fact that the various reducing sugars, dextrose, levulose, and mannose are readily convertible into one another

by the action of lime and many of its compounds. Such a reaction is in fact very marked in an alkaline clarification of cane juice, as by carbonation, and as a consequence the molasses from such juice, as Pellet has shown, contains reducing sugars which were not originally present, such as mannose and glucose.

The extreme sensitiveness of dextrose and levulose to molecular changes in the presence of the merest trace of alkali may be nicely shown by boiling a solution of either sugar in a glass flask for several hours. The alkali dissolved from the glass is sufficient to effect the transformation of a considerable amount of one sugar into the other. Experiments upon the transmutation of sugars usually show a more rapid tendency upon the part of dextrose to pass into levulose than the reverse, and the excess of levulose in low grade cane products may be satisfactorily explained upon this assumption.

ALKALINE CLARIFICATION WITH LIME AND SULPHUROUS ACID.

In this experiment, the juice, after liming to 1 cc. alkalinity, was heated and skimmed; it was then brought back with sulphurous acid to .2 cc. acidity. The remainder of the process of clarification being conducted in the usual way.

As in the previous experiments, when an excess of lime was used, we note a falling off in the glucose content of the clarified juice and an increase in the combined acids and gums. The increase in glucose ratio of the syrup and continued decrease in purity indicate a slight inversion during boiling.

TABLE XXV.

Limed to 1 cc. alkalinity, heated and skimmed.
Sulphured to .2 cc. acidity.

Plant Cane. D. *74.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	16.12	13.62	0.60	0.59	84.49	8.73	0.48	0.09	0.12	0.09	0.096	0.150
Clarified Juice	16.24	13.65	0.60	0.52	84.05	8.20	0.48	0.02	0.19	0.24	0.023	0.152
Syrup	50.00	41.67	1.79	2.01	83.34	9.12	1.53	0.11	0.73	0.77	0.093	0.498
First Massecuite	90.00	77.38	3.43	3.49	(85.98)	6.66	2.76				0.172	0.875
First Sugar		96.00	1.54									
First Molasses	80.00	52.42	6.64	7.41	65.52	26.80	6.39				0.287	1.775
Second Massecuite	90.00	59.55	8.01	8.77	66.17	28.18	7.10				0.262	2.251
Second Sugar		86.61	1.96	2.10								
Second Molasses	80.00	41.14	9.33	10.60	51.42	48.44	9.63				0.355	2.834

Alkaline Clarification with Lime and Carbonic Acid.

Carbonation, as alkaline clarification with lime and carbonic acid is termed, has been applied to the manufacture of sugar from beets with much success, and to a limited extent with sugar canes from tropical countries where the glucose content of the juice is very small.

The comparative immaturity of our sugar canes in Louisiana, and their correspondingly high glucose content, has, as was previously shown, not permitted the economical use of large quantities of lime at the temperatures ordinarily employed.

To this end, a reduction of the temperature and a large increase in the amount of lime was employed. The excess of lime was removed by carbonic acid, sulphurous acid or phosphoric acid in various manners.

The following tables give the methods of conducting the experiments and their results. In all of these the results were calculated to a basis of 12 per cent of sucrose for uniformity in comparison:

TABLE

CARBONATATION EXPERIMENT	Number	Cubic Centimeters Acidity	Cubic Centimeters Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio
CLARIFIER No. 6—										
Mill juice	1	1.05	.....	15.96	15.78	12.00	2.03	1.93	75.19	16.93
Juice limed and heated to 60–68° C.	2	.....	7.90	15.79	14.55	12.00	0.92	2.87	75.97	7.67
Juice limed as in No. 2, carbonic acid passed in, then heated to 90° C. and filtered	3	.....	0.55	15.28	14.48	12.00	0.86	2.42	78.55	7.17
Juice treated as above, then phosphoric acid added to acidity	4	0.45	.....	15.02	14.68	12.00	0.83	2.19	79.89	6.92
CLARIFIER No. 7—										
Mill juice	5	1.00	.....	16.18	15.78	12.00	2.07	2.11	74.16	17.25
Juice limed and heated to 56° C. then filtered	6	.....	21.30	17.65	16.29	12.00	0.72	4.93	67.99	6.00
Juice limed as above, carbonic acid passed in, heated to 90° C. and filtered	7	.....	1.60	15.47	14.23	12.00	0.52	2.95	77.56	4.33
Final clarification of No. 7	8	.....	0.25	15.12	14.12	12.00	0.36	2.76	79.37	3.00
CLARIFIER No. 8—										
Mill juice	9	1.10	.....	15.67	15.27	12.00	1.64	2.03	76.58	13.67
Limed, carbonated and filtered Clarifier No. 8 Lime equivalent to 0.75 % of weight of cane added	10	.....	1.40	14.85	14.24	12.00	0.43	2.42	80.81	3.58
Clarified Juice, Clarifier No. 8—Second carbonatation	11	....	0.15	14.98	13.83	12.00	0.72	2.26	80.11	6.00
Syrup of carbonatation Experiments diluted and reclarified with phosphoric acid—phosphoric acid added to 2.2 cc. acidity and limed	12	0.60	.....	15.54	15.24	12.00	0.76	1.78	77.22	6.33
Mill juice	13	2.50	.....	17.55	17.00	12.00	3.87	1.68	68.38	32.25
Limed juice	14	.....	5.30	16.68	16.11	12.00	2.04	2.64	71.94	17.00
Limed juice after carbonatation	15	.....	0.20	16.70	16.06	12.00	2.48	2.22	71.86	20.67
Juice decanted, boiled and skimmed	16	.....	0.20	16.44	15.76	12.00	2.03	2.41	72.99	16.92

XXVI.

Total Proteids	Albuminoids	Amids	Ash	Lime	Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate	Ash in Alcoholic Precipitate	Lime in Alcoholic Precipitate	Gums, etc.	Phosphoric Acid in Alcoholic Precipitate	Phosphoric Acid in Juice	Sulphurous Acid	Sulphuric Acid
.2690	.1356	.1334	.3610	.0300	.4940	.1340	.1170	.0170	.2430	.....	.....	....	.....
.1673	.0356	.1217	.7700	.4730	1.3570	.0560	.4950	.1560	.8060	.....	.....	....	.....
.1293	.0350	.0943	.5200	.2520	.5250	.0504	.1930	.1360	.2816	.....	.....	.....	.....
.1902	.0310	.1592	.4970	.2230	.3337	.0275	.1736	.0947	.1336	....	.. ...	.....	.. ..
.2905	.1485	.1420	.3680	.0420	.3906	.1350	.1138	.0156	.1418	.....	.....	.....	.....
.1669	.0639	.1030	1.7350	1.1110	2.0402	.0785	.8037	.5661	1.1580	. ...	.....	.....	....
.1328	.0247	.1081	.6190	.3510	.5283	.0250	.1974	.1381	.3059	...	.....	.....	.....
.1356	.0192	.1164	.6080	.3340	.4028	.0213	.1265	.1021	.2550	.....	... .	.....	.....
.2634	.1491	.1143	.4960	.0290	.4524	.1311	.1205	.0116	.2008	.....	...	.....	.....
.1431	.0318	.1113	.5090	.2500	.5595	.0153	.2319	.1729	.3123	.....	....	.....	.....
.1175	.0241	.0934	.5530	.2430	.3540	.0180	.1290	.0750	.2070	. ....	. ..	.....	.....
.1694	.0239	.1355	.6470	.2960	.3718	.0254	.1376	.....	.2088	.....	.....	.....	.....
.3579	.1845	.1734	.4610	.0910	.5122	.1300	.0715	.0910	.3107	.....	.....	.....	.....
.2318	.0428	.1890	.7360	.3830	.8337	.0408	.3515	.2619	.4914	.....	....	.....	.....
.2330	.0430	.1900	.0437	.0410	.2755	.0154	.0701	.0520	.1900	. ...	.....	.....	.....
.1943	.0428	.1515	.4810	.0790	.2346	.0162	.0510	.0372	.1676	.....	....	.....	.....

TABLE XXVI.

CARBONATATION EXPERIMENT	Number	Cubic Centimeters Acidity	Cubic Centimeters Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio
CLARIFIER No. 8—*Continued.*										
Syrup from decanted juice. boiled and skimmed	17	0.35	.....	15.96	15.20	12.00	2.05	1.91	75.19	17.08
Juice boiled with settlings	18	0.55	.. ..	16.78	16.36	12.00	2.33	2.45	71.52	19.42
Mill juice	19	1.40	.....	15.92	15.19	12.00	2.23	1.69	75.38	18.58
Limed juice before heating. Alkalinity with precipitate 24.0 cc. Without precipitate 21.4 cc.	20	. ....	21.50	17.48	16.52	12.00	1.99	3.49	68.65	18.42
Limed juice heated to 56° C	21	.....	16.10	17.48	17.04	12.00	0.90	4.58	68.65	7.50
Phosphoric acid added to juice heated to 56° C. to 0.2 cc. acidity before heating	22	0.10	.....	15.55	15.02	12.00	1.55	2.00	77.17	12.92
Juice of No. 21 carbonated in excess of lime	23	0.35	.....	16.20	15.57	12.00	1.63	2.57	74.07	13.58
Juice of No. 21 carbonated to 2 cc. alkalinity, heated to 85° C, filtered and juice was found to be .5 cc. acid	24	0.50	.....	16.28	15.54	12.00	0.68	3.60	73.71	5.67
Juice of No. 21 sulphured to .5 cc. alkalinity, heated to 85° C, filtered and sulphured till slightly acid, heated to boiling and filtered	25	0.50	.....	16.68	15.56	12.00	0.60	4.08	71.35	5.00
Mill juice	26	1.20	. ...	15.90	15.61	12.00	1.98	1.92	75.47	16.50
Juice limed to 22.1 cc. alkalinity and heated and heated to 30° C..	27	.....	19.80	17.28	15.58	12.00	1.69	3.59	69.45	14.08
Juice limed to 22.1 cc. alkalinity and heated to 40° C.	28	.....	18.80	17.29	15.53	12.00	1.64	3.65	69.40	13.67
Juice limed to 22.1 cc. alkalinity and heated to 50° C.	29	.. ..	18.00	17.39	15.74	12.00	1.49	3.90	69.01	12.42
Juice limed to 22.1 cc. alkalinity and heated to 60° C.	30	.....	15.20	17.07	15.52	12.00	1.07	4.00	70.30	8.92
Juice limed to 22.1 cc. alkalinity and heated to 70° C.	31	... ..	12.80	17.22	15.84	12.00	0.70	4.52	69.69	5.83
Mill juice	32	1.45	.....	16.72	16.57	12.00	2.42	2.15	71.77	20.17

—Continued.

Total Proteids	Albuminoids	Amids	Ash	Lime	Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate	Ash in Alcoholic Precipitate	Lime in Alcoholic Precipitate	Gums, etc.	Phosphoric Acid in Alcoholic Precipitate	Phosphoric Acid in Juice	Sulphurous Acid	Sulphuric Acid
.2241	.0388	.1853	.4980	.0910	.2556	.0160	.0546	.0381	.1894	.....	....	.. ..	.....
.2159	.0412	.1747	.4880	.0850	.2775	.0103	.0435	.0329	.2237	.....	.....	.....	.....
.2710	.1127	.1583	.3726	.0379	.2526	.1105	.6310	.0113	.0790	.0077	.0404	.....	.....
.2254	.0792	.1462	1.6584	.8744	2.4904	.0712	.6752	.6146	1.7440	.....	.....	.....	.....
.1774	.0596	.1178	1.6977	.8895	2.7269	.0695	.6830	.6109	1.9744	.....	... ..	.....	.....
.1406	.0390	.1167	.5909	.1990	.0945	.0348	.0323	.0162	.0274	.....	.1120	...	.....
.1603	.0336	.1267	.6588	.1980	.2076	.0204	.0540	.0442	.1332	....	.0246	....	....
.1613	.0374	.1239	.6580	.2390	.2730	.0291	.0741	.0638	.1698	.....	.....	.....	.....
.1521	.0115	.1406	.7242	.3024	.2424	.0188	.0688	.0552	.1548	.0031	.0092	.0768	.1536
.2318	.1168	.1150	.2751	.0339	.1528	.0953	.0429	.0124	.0136	... ..	.....	.....	.....
.1930	.0745	.1185	.9200	.7293	1.9300	.0705	.5374	.4420	1.3221	....	.....	.....	.....
.1595	.0614	.0981	.8722	.7053	2.1394	.0511	.6138	.4229	1.4745	... ..	.....	.....	..... .
.1738	.0668	.1070	.9200	.7293	2.0039	.0449	.6552	.5475	1.3038	.....	....	.....	.....
.1515	.0539	.0976	.9138	.7156	1.7661	.0539	.5197	.3809	1.1915	.....	....	.....	.....
.1691	.0555	.1046	1.1288	.7088	1.4776	.0556	.4628	.4040	.9592	.. ..	.....	.....	.....
.2275	.1151	.1124	.2557	.0485	.2457	.0510	.0412	.0036	.1535	.....	.....	.. ..	.....

TABLE XXVI.

CARBONATATION EXPERIMENT	Number	Cubic Centimeters Acidity	Cubic Centimeters Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio
CLARIFIER No. 8—*Continued.*										
Juice limed to 20 cc. alkalinity and heated to 40° C.....	33	.....	17.80	17.94	16.40	12.00	1.88	4.06	66.95	15.67
Juice limed to 20 cc. alkalinity and heated to 45° C..... ..	34	.....	16.40	17.85	16.11	12.00	1.75	4.10	67.23	14.58
Juice limed to 20 cc. alkalinity and heated to 50° C.....	35	.....	16.80	17.76	15.60	12.00	1.63	4.13	67.57	13.58
Juice limed to 20 cc. alkalinity and heated to 55° C.....	36	.....	17.40	18.27	16.49	12.00	1.43	4.84	65.68	11.75
Mill juice.	37	2.30	.....	21.62	21.09	12.00	5.09	4.53	55.50	42.42
Juice sulphured to 7.3 cc. acidity and limed to .05 cc. alkalinity	38	.....	0.05	20.92	20.24	12.00	4.59	4.33	57.37	38 25
Juice limed to 10.5 cc. alkalinity, heated to 45° C. and sulphured to 0.4 cc. alkalinity...	39	.....	0.40	19.58	18.37	12.00	3.84	3.74	61.24	32.00
Juice limed to 10.5 cc. alkalinity, heated to 45° C. and phosphoric acid added.....	40	.....	0.40	19.46	18.88	12.00	3.84	3.62	61.66	32.00
Juice limed to 10.5 cc. alkalinity, heated to 45° C, carbonated and filtered, then heated to 95° C	41	.....	0.35	20.20	18.78	12.00	3.78	4.42	59.41	31.50

—Continued.

Total Proteids	Albuminoids	Amids	Ash	Lime	Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate	Ash in Alcoholic Precipitate	Lime in Alcoholic Precipitate	Gums, etc.	Phosphoric Acid in Alcoholic Precipitate	Phosphoric Acid in Juice	Sulphurous Acid	Sulphuric Acid
.1986	.0704	.1182	1.7595	1.1662	2.1348	.0720	.7128	.6288	1.3500	.....	.....	.....	.....
.1477	.0752	.0725	.9373	.....	2.7227	.0752	.7015	.5853	1.9460	.....	.....	.....	.....
.1710	.0640	.1070	1.0090	.8269	2.6860	.0635	.6760	.5880	1.9465	.....	.....	.....	.....
.1725	.0735	.0990	1.3242	.9166	2.6797	.0735	.7200	.5101	1.8862	.....	.....	.....	.....
.4000	.2165	.1835	.5090	.....	1.2362	.1024	.1483	.0214	.9855	.....	.....	.....	.....
.3834	.1211	.2623	.6280	.1230	.8170	0750	.1800	.0980	.5620	.....	.....	.....	.....
.3666	.0887	.2779	.5095	.1019	.7670	.0610	.0885	.0670	.6175	.....	.....	.....	.....
.3279	.0893	.2486	.4875	.1035	.5530	.0218	.0994	.0574	.4318	.....	.....	.....	.....
.....	.0997	.....	.4949	.0841	.7295	.0526	.0841	.0447	.5928	.....	.....	.....	.....

On examining the above tables, it will be noted that there are several stages in each clarification showing the action of the clarifying agents at these points. Numbers 1 to 4, inclusive, which are the results of liming strongly and single carbonating, show the action of the excess of lime. Carbonic acid precipitates the lime in No. 3, while No. 4 is the result of the substitution of phosphoric acid for carbonic acid. From an ordinary analysis including total solids, sucrose and glucose, all results show marked improvement over the mill juice. It is noted, however, that in all except the phosphoric acid clarification, there is an excess of gums, etc., in the clarified over the raw juice. In the phosphoric acid clarification where nearly fifty per cent of the gums of the mill juice are removed, a large amount of combined lime remains, which was found to prevent the concentration of the juice in the vacuum pan. Examining 5 to 8, embracing one clarification with double carbonating to remove the excess of lime, an improvement over the limed juice as in the preceding single carbonation is noted, but even the final clarification shows these juices to contain more impurities than are in the mill juice.

Here, during the time the lime was in large excess, the temperature was not above 68° Centigrade. Notwithstanding this fact, the injury was due to the action of this excess of lime on the glucose. This is shown by the destruction of glucose and the large increase in the amount of gums. The same deleterious results are noted throughout the tables, the excess of lime so injuring the character of the juice that it could not by any practical means be brought into such a condition as to render it fit for manufacturing purposes.

Further examination of the tables, Nos. 19 to 25, inclusive, shows a combination of the uses of lime, sulphur, phosphoric and carbonic acids in an attempt to overcome, if possible, the evil of the action of the lime at low temperatures. A close perusal of the results will show that while these agents removed a considerable quantity of impurities, in no instance was even the purity increased. The glucose shows considerable variation. The result of heating the alkaline juice and the quantity of lime remaining is evidence that the lime combined with organic acids. This is fully shown by the large increase in the amount of gums, acids, etc., as well as by the fact that the clarified juices are only

slightly acid and this acidity is not sufficiently great to account for the large quantity of organic acids remaining in solution. Phosphoric acid, No. 22, gave the best clarification, and the clarified juice is left nearly neutral (0.1 cubic centimeter acid), but in this the unusually large amount of lime remaining in solution combined with organic acids, hinders evaporation and crystallization.

Numbers 26 to 31, inclusive, give the mill juice compared with the juice limed cold to 22.1 cubic centimeters alkalinity, then heated at ranges of 10° Centigrade, starting at 30° Centigrade and ending at 70° Centigrade. The alkalinity decreases as the temperature increases. The purity of all clarifications is considerably reduced when determined by the Brix, but the true purity determined by the actual total solids varies very little from that of the mill juice. The glucose decreases with the elevation of the temperature, and the solids not sugar increase. The albuminoids decrease, being broken up in the 40°, 50°, 60° and 70° Centigrade clarifications into amids which by the strong alkalinity and high temperature are partially converted into ammonia and amido acids; the total amids, however, remain the same. The ash shows very little fluctuation, as does the lime remaining in solution. The gums increase in quantity up to 40° Centigrade, then decrease from the maximum as the temperature is raised, as the action of the increasing temperature changes their composition.

Taking the comparison as a whole, the alkalinity was less injurious at 30° to 40° Centigrade than at the other temperatures. At this point its action on the glucose and amids is the least destructive and it forms the smallest amount of salts of organic acids, which are more objectionable than the original compounds.

Numbers 32 to 36 compare the same clarification with a large quantity of lime alone (20 cubic centimeters alkalinity), at ranges of 5° in temperature for the clarification.

Comparing these, it will be noticed that No. 33 gave the best results and the temperature here was 40° Centigrade, confirming previous results, where 30° to 40° was found most beneficial. Numbers 37 to 41, inclusive, are clarifications of a fermented juice by treating with sulphur followed by liming to practical neutrality. The lime was followed by sulphur in No. 39, by phosphoric acid in No. 40, and by carbonic acid in No. 41. Here in No. 40 it is seen that phosphoric acid, followed by sulphurous acid, gives the best clarification, but in none of these clarifications can any marked improvement in the clarified juice be noted, while the large amount of lime remaining in solution is very objectionable.

None of these strongly alkaline clarifications at low temperatures were found as beneficial as the methods at present adopted in the sugar houses. The excess of lime, except where heat was only 30° to 40° Centigrade (and unfortunately this clarification was not completed), in every instance attacked the glucose strongly, and the products formed were very injurious. Where concentration was attempted it was found to darken the juice considerably, while in attempting to evaporate to masse cuite the lime salts coated the coils and so retarded the circulation of heat that evaporation beyond a thick syrup was impossible.

Noting the injuries caused by the use of lime in large quantities, even at low temperatures, and the inefficiency of either carbonic, sulphurous or phosphoric acids in correcting these injuries, it was thought that by using a smaller excess of lime more satisfactory results could be obtained.

The following table shows the result of the sulphur and lime clarifications, the juice while alkaline being heated to 55° Centigrade, instead of 99°, as in ordinary clarifications:

TABLE XXVII.

	Acidity	Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio	Total Proteids	Albuminoids	Amids	Ash	Lime	Sulphurous Acid (SO_2)	Sulphuric Acid (SO_3)	Alcohol Precipitate	Albuminoids in Alcohol Precipitate	Ash in Alcohol Precipitate	Lime in Alcohol Precipitate	Gums, etc.
Mill Juice, Clarifier No. 51.	1.45	....	16.58	15.91	12.00	2.00	2.58	71.22	16.67	.4006	.1624	.2382	.485	.061	.....	.....	.3367	.1488	.1105	.0126	.0774
Limed Juice, Clarifier No. 51—Lime added and heated to 55° C.....	....	2.9	16.52	16.35	12.00	2.20	2.32	72.63	18.33	.2681	.0564	.2117	.597	.196	.....	.....	.5595	.0356	.2328	.1592	.2911
Limed Juice, Clarifier No. 51—Sulphured to acidity and clarified. During sulphuring the temperature rose to 78° C.....	1.05	....	16.43	15.86	12.00	2.05	2.38	73.04	17.08	.2494	.0472	.2022	.609	.149	.060	lost	.2570	.0197	.0992	.0552	.1381

These results show that even at this low temperature, with lime in excess (2.9 cubic centimeters alkalinity), the glucose was not attacked as in the clarifications where higher temperature was employed. The purity increased 1.41 per cent by Brix, and to a larger extent by the actual solids. The amids, however, were attacked and the gums largely increased over those in the mill juice. The sulphuring of this juice to acidity (1.05 cubic centimeters), while removing a considerable quantity of the impurities, did not give as pure a juice as that started with, and this method of clarification must therefore be condemned.

The results of the substitution of phosphoric acid for sulphurous acid, then varying the alkalinity and the amount of phosphoric acid added, are given in Table XXVIII.

On examining Table XXVIII it was found that phosphoric acid proved to be very beneficial in its results. The final clarifications, both when phosphoric acid was added to slight acidity and when the juice was left slightly alkaline, showed very good results.

The action of lime alone at the temperature of 55° to 57° Centigrade shows the same effects as those found in all strongly alkaline clarifications. The glucose is attacked, gums, etc., are increased, and the ash and lime which remain in the juice are also increased. The action of phosphoric acid on this partially clarified juice is very beneficial. Without the application of heat it gives a large increase in purity, restoring the glucose to nearly its original percentage. It further removes the albuminoids and largely removes the gums, the amount of gums remaining in this juice being considerably less than in the mill juice. Continuing with this clarification by heating No. 3 until the conditions of normal clarification are reached, the benefit of the phosphoric acid is still maintained, though a very small amount of ash, gums, etc., is brought into solution. The purity is apparently lowered, but if the true total solids be examined, it is increased. No. 5 is the clarified product from the same mill juice; in which phosphoric acid has been added in quantity not sufficient to neutralize the lime. The excess in the previous clarification was only a trace (.05 cubic centimeter acidity). Here the clarified juice was left slightly alkaline, the alkalinity being .1 cubic centimeter. In this clarification the glucose was not attacked to an

TABLE XXVIII.

	Number	Acidity	Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio	Total Proteids	Albuminoids	Amids	Ash	Lime	Phosphoric Acid (P_2O_3)	Alcoholic Precipitate	Albuminoids in Alcohol Precipitate	Ash in Alcohol Precipitate	Lime in Alcohol Precipitate	Gums, etc.
Raw Juice	1	1.05	....	16.70	16.21	12.00	2.49	2.21	71.86	20.75	.3220	.1452	.1768	.4066	.0377	.0269	.3846	.1169	.1835	.0226	.0842
Limed Juice, 20 lbs. lime added to 350 gallons of juice and heated to 55-57° C.	2	....	6.30	17.55	16.47	12.00	1.85	3.70	68.38	15.42	.2062	.0454	.1608	.813	.436	.....	1.0635	.0408	.8975	2827	.1252
Clarified Juice, Phosphoric Acid—added to slight acidity before heating	3	0.05	....	15.21	14.95	12.00	1.97	1.24	78.89	16.42	.1790	.0325	.1465	.365	.048	.008	.0480	.0029	.0180	.0072	.0271
Clarified Juice—After heating	4	0.05	.. .	15.44	14.84	12.00	1.92	1.52	77.72	16.00	.1801	.0276	.1525	.389	.042	.007	.0648	.0031	.0300	.0144	.0317
Clarified Juice, Clarifier No. 37—Mill juice treated as in No. 36	5	....	0.10	15.39	14.99	12.00	2.40	0.99	77.97	20.00	.1650	.0268	.1382	.363	.090	.005	.0163	.0011	.0110	.0034	.0042

appreciable extent, while the purity was raised to about the same as that in the acid clarification (No. 4). The gums were removed in greater amount, the total alcoholic precipitate being less than the gums which remained in the alkaline clarification. The gums, etc., are almost all removed from the clarified juice (No. 5), only .0042 per cent remaining.

This shows that by employing a low temperature with a moderate amount of lime, as compared to carbonatation, or a large excess as compared with normal clarifications, then removing this excess by phosphoric acid, a very large part of the impurities are removed and the results are beneficial.

ACID CLARIFICATION.

In the previously discussed chemical clarifications, the juices were treated with lime to the points of neutrality or alkalinity before beginning the removal of impurities. We will now take up a series of clarifications of an exactly opposite type, where the juice is first acidified with either phosphoric or sulphurous acids.

The great danger of an alkaline clarification consists in the formation of gums and other melassegenic products. An equally great risk—that of the inversion of sucrose—is incurred in an improperly conducted acid clarification. The beautiful clarification which a liberal use of phosphoric or sulphurous acids effect is well known to every sugar maker. Bright, limpid juices are obtained which are free from gums and which can be evaporated and boiled with the greatest ease, yielding masse cuites which give a prime quality of first sugars with no difficulty in purging. When sulphitation was first introduced in Louisiana, the careless sugar maker was frequently content with this apparent smoothness of operations, and paid no attention to the excessive losses of sucrose, which resulted in yields as low as 90 pounds of sugar per ton of cane.

ACID CLARIFICATION WITH PHOSPHORIC ACID AND LIME.

This method is usually found to give very satisfactory results, but it is practiced in Louisiana to only a limited extent, the relatively high cost of phosphoric acid being the great obstacle in the extension of this process.

In the experiment, Table XXIX, the juice was acidified with phosphoric acid to five cubic centimeters N-10 acidity, and after bringing back with lime to 0.2 cubic centimeters acidity, was heated and worked in the usual way.

It will be noted that a higher degree of exhaustion is attained in the second molasses than is the case in any of the experiments with alkaline clarification. A slight inversion seems to have taken place in the boiling of the second masse cuite, as is indicated by the increase in glucose ratio and the decrease in purity. As regards the changes taking place in the ratio of dextrose to levulose and of albuminoids to amids during the process of manufacture, the same observations hold as were noted in previous experiments.

Acid Clarifications with Sulphurous Acid and Lime.

This method of clarification, known as sulphuration or sulphitation, is the one most commonly practiced in Louisiana, being used in over 80 per cent of the sugar houses. The general practice is to sulphur the juice to from 3 cubic centimeters to 5 cubic centimeters N-10 acidity for 10 cubic centimeters of juice; after bringing back with lime to faint acidity or neutrality the juice is heated until the blanket of impurities begins to break. It is then either skimmed and allowed to settle in the clarifier or else run into settling tanks, where the suspended impurities are allowed to deposit. Separating the juice from scums and sediment before settling is frequently practised, this being found to assist in the mechanical elimination of impurities. After leaving the settling tanks, which work automatically, the juice in some sugar houses in further purified by passing through bag filters.

Sulphur in Combination with Lime and Heat as Clarifying Agents.

Before entering into results of the use of sulphur in clarifying cane juice, a short statement of its properties will be given.

When sulphur is burned in the air it unites with oxygen, forming a gas called sulphur dioxide. This, on being forced into cane juice, combines with water to form sulphurous acid. One pound of sulphur forms two pounds of sulphur dioxide. Sulphur dioxide is quite soluble in cane juice, one gallon of juice at ordinary temperature and pressure dissolving 33 gallons of sul-

TABLE XXIX.

Run 7. Phosphoric Acid to 5cc. acidity.
Lime to .2cc. acidity.

Plant Cane. D. 74.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	15.49	12.85	0.74	0.65	82.85	10.82	0.45	0.08	0.12	0.13	0.097	0.127
Clarified Juice	15.21	12.77	0.65	0.58	83.96	9.63	0.44	0.02	0.16	0.13	0.025	0.124
Syrup	50.00	41.43	2.04	2.38	82.86	10.67	1.51	0.11	0.60	0.50	0.108	0.493
First Masse cuite	90.00	78.13	3.51	4.39	86 81	10.11	2.58				0 160	0.796
First Sugar		95.60	2.00									
First Molasses	80.00	51.40	7.1	7.56	64.25	28.54	5.43				0.249	1.621
Second Masse cuite	90.00	59.71	8.52	10.33	66.34	31.57	6.11				0.257	1.817
Second Sugar		81.89	2.88	3 57								
Second Molasses	80.00	32.45	11.37	13.38	40.56	76.28	7.40				0.324	2.580

phur dioxide, or the amount of sulphur dioxide formed from 6.33 ounces of sulphur. It is therefore possible to use 6.33 ounces of sulphur to every gallon of juice. This amount of sulphur dioxide, however, cannot be made to combine with the juice in ordinary sugar house work, as it is quite difficult to cause cane juice to absorb its full capacity of this gas. In practice it is difficult to use more than .2146 ounce of sulphur dioxide or .1073 ounce of sulphur per gallon of cane juice.

At high temperatures all sulphur acids invert cane sugar very strongly. In its inverting effects sulphurous acid ranks second among the sulphur acids, and fourth in the entire list of acids.

Experiments on the inverting power of sulphurous acid, as ordinarily used in clarification, showed that at the ordinary temperature no inversion took place when the juice was allowed to remain acid for a moderate time, but when heated to 150° Fahrenheit 23 per cent of the sucrose was inverted in one-half hour, and in one hour at 195° Fahrenheit practically all of the sucrose was inverted. The acidity of the juice was equivalent to a twentieth normal solution of sulphurous acid. Sulphur dioxide when absorbed by cane juice raises the density in proportion to the amount absorbed and shows a corresponding decrease in purity when the Brix spindle or saccharometer is used to estimate the solids. This, however, is only temporary, as on the addition of lime, sulphites of lime are formed, which are comparatively insoluble in the juice, and remove the sulphurous acid. Sulphurous acid also precipitates some of the albuminoids and exercises a bleaching effect on the coloring matters of the juice.

Before giving the results, a further explanation of the terms used and their equivalents will be given. The acidity or alkalinity of these juices is determined by means of a tenth (1-10) normal acid or alkali solution, using either lime, sodium or potassium hydrate as the alkali, with phenolphthalein as an indicator, and is expressed in terms of the number of cubic centimeters of solution required to neutralize 10 cubic centimeters of juice. One cubic centimeter acidity is equivalent to .02146 ounce of sulphur dioxide per gallon and one cubic centimeter alkalinity is equivalent to .0375 ounce of lime per gallon.

Table XXX shows the action of sulphurous acid, lime and heat in clarifying.

In this table it will be noticed that the sulphuring was carried to the same point in each clarification, the amount of lime added varying with each clarification, except in No. 9 and No. 11.

The sulphured juices show a decrease in purity when calculated on the degree Brix and even when calculated on the total solids, though in the latter case the differences are considerably less, except in one instance. This decrease is caused by the higher specific gravity of the sulphurous acid dissolved in the juice, and as most of this is driven off during evaporation, a lesser increase in total solids, when determined by this method, is noted.

In No. 1 to No. 3, inclusive, the analyses of the mill, sulphured and clarified juices are given. Clarification has also been made on a juice with a large quantity of the acids of the juice and some sulphurous acid left unneutralized by lime, the acidity being 3.0 cubic centimeters. In this experiment the purity of the clarified juice is not equal to that of the mill juice. The increase in glucose in the sulphured juice is due to the length of time it was allowed to remain so strongly acid (10 cubic centimeters) before the analysis was made. Not showing in the clarified juices, it is evident that the inversion did not take place before clarification. In this clarification there is very little variation in the solid and sugar contents of the mill and clarified juices. There is, as expected from the acidity of the juice, a considerable quantity of sulphur in the form of free sulphurous and sulphuric acids which remain, in the clarified juice. Seventy-four per cent and 74.6 per cent, respectively, of the gums, etc., and albuminoids are removed.

In the next clarification (Nos. 4 to 6, inclusive) sulphuring was carried on to the same point and the juice was limed until the acidity was reduced to 1 cubic centimeter. Quite an increase in purity is noted in the clarified juice when calculated on the Brix. This, however, is only apparent, due doubtless to mineral bodies, the specific gravities of whose solutions are higher than the specific gravities of sucrose solutions containing the same per cent of solid matter. By actual solids there is very little

TABLE XXX.

	Acidity	Alkalinity	Total Solids	Brix	Sucrose	Glucose	Solids Not Sugar	Purity Coefficient	Glucose Ratio	Sulphurous Acid	Sulphuric Acid	Lime	Ash	Total Proteids	Albuminoids	Amids	Alcohol Precipitate	Gums, etc., in Alcohol Precipitate	Ash in Alcohol Precipitate	Albuminoids in Alcohol Precipitate	Lime in Alcohol Precipitate
1. Mill juice	.....	....	.....	15.27	12.00	1.74	1.53	78.59	14.50	...	....	...	....	.208	.097	.111	.361	.213	.040	.168	....
2. Sulphured juice	10.00	....	.....	15.96	12.00	1.84	2.12	75.19	15.33	.312	.013	....	....	.219	.098	.121	.401	.226	.052	.123	.012
3. Clarified juice	3.00	...	.....	15.37	12.00	1.75	1.62	78.07	14.58	.079	.082	....	....	.138	.025	.113	.241	.054	.176	.011	.106
4. Mill juice	.....	....	15.17	15.82	12.00	2.00	1.83	75.92	16.66	..	....	....	.578	.232	.126	.107	.270	.156	.022	.092	.003
5. Sulphured juice	10.00	....	15.34	16.14	12.00	2.04	2.10	74.32	17.00	.305	.090	....	.507	.232	.127	.105	.370	.226	.051	.093	.006
6. Clarified juice	1.00	....	15.32	15.55	12.00	2.02	1.53	77.17	16.84	.071	.095	.168	.403	.145	.026	.119	.057	.051	.003	.003	.001
7. Mill juice	.....	....	14.38	15.12	12.00	1.44	1.68	79.36	12.00	....	....	....	.598	.188	.100	.088	.334	.227	.027	.080	.006
8. Sulphured juice	10.00	....	14.57	15.00	12.00	1.34	1.66	80.13	11.15	.203	.045	....	.633	.180	.100	.080	.341	.208	.048	.085	.017
9. Clarified juice	0.00	0.00	14.19	14.83	12.00	1.38	1.45	80.92	11.50	.058	.049	.048	.206	.104	.015	.089	.037	.032	.001	.004	.000
10. Mill juice	.....	....	14.02	14.22	12.00	1.00	1.22	84.39	8.33	....	....	....	.336	.126	.084	.042	.279	.170	.034	.075	.003
11. Sulphured juice	10.00	...	13.93	14.40	12.00	1.05	1.35	83.33	8.75	.202	.000		.285	.120	.075	.045	.261	.167	.027	.067	.004
12. Clarified juice	0.00	0.00	14.07	14.16	12.00	1.02	1.14	84.75	8.50	.033	.020	.035	.225	.060	.014	.046	.039	.029	.002	.008	.000
13. Mil juice	.....	....	15.23	15.61	12.00	1.74	1.87	76.87	14.50	....	....	....	.461	.309	.133	.176	.371	.185	.042	.044	.006
14. Sulphured juice	10.00	....	15.28	15.66	12.00	1.73	1.93	76.63	14.42	.250	.057	....	....	.280	.125	.155	.372	.163	.106	.103	.013
15. Clarified juice	....	1.00	15.09	15.40	12.00	1.63	1.77	77.92	13.59	.046	.023	.052	.541	.223	.032	.190	.058	.044	.007	.007	.005

difference in purity. The sulphur salts remain in solution in large proportions, almost equaling those in the more strongly acid juice (No. 3). The amount of lime left in solution as lime salts is .168 per cent of the juice. Thirty and three-tenths per cent of the ash, 79.4 per cent of the albuminoids and 67.3 per cent of the gums, etc., present in the mill juice are removed. This, while not as large a percentage as in the clarification No. 3, leaves less gums in the juice on account of its being from purer cane, for, as noted under the results of juice extracted by the different mills (at different pressures), the more impure the juice the greater is the action of clarifying agents, both in quantity and percentage. Continuing the increase of lime to the neutral point, there are given the results of two clarifications, No. 9 and No. 12. In both instances an increase in purity is noted over that of the mill juice, the purer juice, as is expected, showing the smaller increase in purity. The salts of the sulphur acids are removed in larger quantity, and in No. 12 very little of such compounds remains in solution. The amount of lime remaining in solution is considerably reduced, being .168 in the 1. cubic centimeter acid clarification and .048 and .035 in the neutral clarifications. The amount, as well as the percentage, of ash removed is higher than in the previous clarifications, but here the more impure juice, No. 9, leads both in amount and percentage. Of the ash present in the mill juice, 64 per cent in No. 9 and 49.3 per cent in No. 12 is removed. The albuminoids removed are 85 per cent and 83.3 per cent, respectively, while the removal of the gums is 85.9 per cent and 82.9 per cent.

Here attention is again called to the benefits derived from a neutral clarification, as both the percentage and amount of impurities removed are largely in excess of the more acid clarifications. Both vary with the purity of the juice, for even in a very pure juice (purity 84%) the percentage of impurities removed is higher than in the clarifications that are allowed to remain acid. Continuing the addition of lime in No. 15 until the juice is made 1 cubic centimeter alkaline, there is an increase in purity again noted. The glucose, however, is slightly attacked and its amount reduced. The evil effects of this were mentioned under lime clarifications and are the same here. The sulphur acids and their salts remaining in solution are more than in the

neutral clarification, but considerably less than the amount remaining in the acid clarification. The lime remaining in solution is, as would be expected, in larger quantity than that present in neutral clarifications, as its excess is the cause of the alkalinity. The ash, instead of decreasing, shows here an increase. Of the albuminoids, 75 per cent are removed. The amids here for the first time in this table show an increase, which, while slight, should be a reminder of the action of the excess of lime on both the albuminoids precipitated and those remaining in the juice. The gums, while largely removed, remain in greater quantity than in the neutral clarifications. In this clarification, 76 per cent of those present in the mill juice are removed, while 85.9 per cent and 82.9 per cent were removed by neutral clarification of juices of higher purity. Where conditions are the same a smaller percentage is removed from juices of higher purity.

From this it will be seen that as with lime alone, sulphurous acid in combination with lime gives the most effective clarification when juices are worked at neutrality. Further study of the action of sulphur and lime as clarifying agents was made; varying amounts of sulphur and lime were used, but each time bringing the juice near the neutral point.

In No. 1 and No. 2 the juice was sulphured with 2.45 cc. of sulphur dioxide, this amount being sufficient to bring the acidity to 3.75 cubic centimeters. Sufficient lime was then added to leave this juice with an acidity of 0.45 cc. Here we note very little improvement in the clarified juice over the mill juice. The purity of the clarified juice according to the Brix spindle is very slightly decreased, but the true percentage of solids shows an increased purity. The albuminoids are largely removed, while the amids and ash are unaffected, the removed mineral matters being substituted by the lime salts of sulphurous and sulphuric acids. The percentage of lime remaining in solution is considerably greater than that in the mill juice. The gums are slightly increased, though the extremely small quantity in this juice would so influence the quantity and percentage removed as to permit no comparison with the juices where the gums are present in average or larger quantities.

In No. 3 and No. 4 the mill and clarified juices are compared, using an increased amount of sulphur over No. 1 and No. 2

TABLE XXXI.

SULPHUR	Acidity	Alkalinity	Brix	Total Solids	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio	Total Proteids	Albuminoids	Amids	Ash	Lime	Sulphurous Acid (S O2)	Sulphuric Acid (S O3)	Alcohol Precipitate	Albuminoids in Alcohol Precipitate	Ash in Alcohol Precipitate	Lime in Alcohol Precipitate	Gums etc.
1. Mill Juice	1 30	....	15.33	15.31	12.00	1.59	1.74	78.28	13.25	.2383	.1426	.0960	.388	.025	....	....	.2230	.1104	.0950	.0135	.0176
2. Clarified Juice, sulphured to 3.75 cc. acidity and limed to 0 45 cc. acidity	0.45	....	15.40	15.01	12.00	1.61	1.79	77.92	13.42	.1371	.0387	.0984	.372	.080	0.94		.089	.017	.040	.0264	.0220
3. Mill Juice	0.95	....	16.26	15.43	12.00	2.14	2.12	73.74	17.83	.2686	.1183	.1503	.423	.040	....	....	.5904	.1091	.1625	.0200	.2189
4. Clarified Juice, sulphured to 5.6 cc. acidity and limed and left slightly acid	0.05	....	15.91	15.00	12.00	2.18	1.73	75.42	18.17	.1444	.0271	1173	.422	.060	....	....	.1955	.0097	.1200	.0378	.0685
5. Mill Juice	0.95	....	15.62	15.25	12.00	1.69	1.93	76.82	14.08	.1718	.0973	.0845	.435	.038	....	....	.3897	.1482	.1129	.0165	.0586
6. Clarified Juice, limed and sulphured as in No. 4	0.05	...	15.15	14.90	12.00	1.85	1.30	79.21	15.42	.1203	.0350	.0853	.344	.048	....	....	.2009	.0181	.1469	.436	.0359
7. Mill Juice	1.90	....	16.20	15.81	12.00	1.94	2.26	74.07	16.17	.2067	.1286	.0781	.542	.035	....	....	.3033	.1072	.0706	.0085	.1255
8 Clarified Juice, limed to equivalent of 5.0 cc. alkalinity and sulphured till slightly acid	0.40	....	16.19	15.51	12.00	1.99	2.20	74.12	16.58	.0948	.0171	.0777	.562	.077	.041	.108	.1663	.0079	.0909	.0385	.0677

and liming to very slight acidity, or, it may be said, practically to neutrality. The juice was sulphured to a total acidity of 5.6 cubic centimeters, and then limed to .05 cubic centimeter acidity.

The purity is increased 1.68 per cent over that of the mill juice, otherwise the sugar analysis is normal, the slight variation in glucose and glucose ratio being well within the limits of analytical error. Seventy-seven per cent of albuminoids present in the mill juice were removed, the ash was practically unchanged and 69 per cent of the gums were removed. Nos. 5 and 6 are a duplication as regards the residual acidity, sulphur and lime used, with the exception of liming first, heating till blanket broke, removing blanket and sulphuring till the juice was brought to an acidity of .05 cubic centimeter. Here an increase in purity of 2.39 is noted with a slight increase in glucose, due to sulphuring while the juice was hot, notwithstanding the fact that the juice was alkaline. When sulphurous acid gas entered the juice it neutralized the lime at the points of contact, causing a local acidity which at high temperatures exercised an inverting influence. There were removed 21 per cent of ash present, 63 per cent of albuminoids, and 39 per cent of gums. Comparing with sulphuring followed by lime, we find a slight increase in purity in favor of liming first, but the gums are removed in greater quantity and percentage by sulphuring first. It may be remarked that the amount of gums is much smaller in mill juice No. 5 than in No. 3. However, in No. 5 there is a considerable quantity and the difference in percentages removed is very great, 69 per cent in No. 3 and 39 per cent in No. 5 being eliminated. Continuing further with liming, using more lime as in clarified juice No. 6, there is no increase in purity by Brix, though the true solids show an increase. The glucose remains nearly the same, and no indication of inversion is seen. Here the ash is slightly increased. The sulphuring was to slight acidity and brought into solution some of the precipitated mineral matter, and in this respect this juice is very similar to clarified juice No. 2, both having the same acidity and nearly the same ratio between their ash contents and those of the mill juices. Of the albuminoids 86.7 per cent were removed, along with 46 per cent of the gums. This increase in the removal of the albuminoids is largely due to bringing the juice to acidity.

The amount of gums removed is small in comparison with the practically neutral clarification using sulphur first. Taking into consideration the difference in the amounts present in juices No. 5 and No. 7, the removal of gums is relatively close to the percentages removed by both clarifications where lime was added first.

From the two foregoing tables it is evident that the best sulphur-lime clarification is obtained by sulphuring strongly, then liming to neutrality.

COMPARISONS OF THE CLARIFIED PRODUCTS FROM COMBINATIONS OF LIME AND SULPHUR ON THE SAME JUICES.

In the following table these comparisons are brought out so as to eliminate the variations caused by cane juices differing in the amount and character of impurities present:

Examining Table XXXII, the first two results given are from the same mill juice sulphured to the same point (5 cubic centimeters). No. 1 was limed until the juice remained slightly acid, and No. 2 was limed until the juice was slightly alkaline. Both were then clarified by heating in the same manner. These results are rather peculiar, and unfortunately the amount of acidity and alkalinity in the clarifications was not determined; though from the results it was thought that No. 2 was left practically neutral. The purity of No. 1 is greater than No. 2; other variations in the sugar and solid content are not marked enough to comment on, taking the regular sugar analysis as a guide. However, in examining the impurities left, it will be noticed that more albuminoids, ash and gums remain in the acid than in the alkaline clarification. In the acid clarification the albuminoids are nearly four times the quantity of those in the alkaline clarification, the ash nearly one-fourth more, and the gums nearly double.

Nos. 4 and 5 are clarified juices from the same cane. No. 4 was limed to 1.5 cubic centimeters alkalinity and then clarified as in lime clarifications, and this clarified juice, without settling, was sulphured to 1.0 cubic centimeter acidity in No. 5, and again heated. Both clarifications show an increase in purity over the mill juice No. 3. The limed clarification showed an increase

TABLE XXXII.

	Number	Acidity	Alkalinity	Total Solids	Brix	Sucrose	Glucose	Solids Not Sugar	Coefficient of Purity	Glucose Ratio	Sulphurous Acid	Sulphuric Acid	Lime	Ash	Total Proteids	Albuminoids	Amids	Alcoholic Precipitate	Gums, etc., in Alcoholic Precipitate	Ash in Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate	Lime in Alcoholic Precipitate
Sulphured to 5 cc. acidity. Lime added, leaving slightly acid	1	....	....	14.08	14.87	12.00	1.44	1.43	80.70	12.00	.1013	.....	.0502	.5620	.2820	.1020	.1800	.2580	.1610	.0360	.0610	.0005
Sulphured to 5 cc. acidity. Lime added to slight alkalinity	2	....	....	14.30	15.35	12.00	1.41	1.94	78.11	11.75	.0550	.....	.0670	.4150	.1890	.0280	.1610	.1060	.0880	.0160	.0020	.0080
Mill Juice	3	....	....	15.20	15.71	12.00	1.59	2.12	76.42	13.25	.....	.....	.....	...	.3620	.1150	.2470	.2670	.1260	.0420	.0990	.0060
Juice limed to 1.5 cc. alkalinity before sulphuring	4	...	1.50	15.10	15.57	12.00	1.47	2.10	77.07	12.25	.....	.....	.1940	.5090	.2580	.0430	.21 0	.3220	.2360	.0450	.0410	.0280
Clarified Juice sulphured to 1 cc. acidity	5	1.00	....	14.03	15.00	12.00	1.37	1.63	80.00	12.00	.0740	.0450	.1750	.4750	.2300	.0370	.1930	.2710	.2170	.0360	.0180	.0320
Juice limed to 3.3 cc. alkalinity before sulphuring	6	....	3.30	14.89	15.76	12.00	2.06	1.55	76.42	17.18	.....	... .	.2760	.5780	.2330	.0300	.2030	.4010	.3350	.0320	.0340	.0240
Clarified Juice of No. 6 sulphured to 2.7 cc. acidity with settlings	7	2.70	....	14.69	15.66	12.00	1.54	2.12	76.62	12.83	.2720	.0010	.2710	.7250	.2250	.0240	.2010	.2480	.1280	.1090	.0110	.0640
Clarified Juice of No. 6 sulphured to 2.7 cc. acidity after removal of settlings	8	2.70	...	15.30	15.80	12.00	1.79	2.01	75.95	14.93	.1410	.0120	.2280	.5960	.2400	.0240	.2160	.2410	.1970	.0350	.0090	.0320

of 65 per cent and the limed and sulphured clarification, No. 5, an increase of 3.58 per cent over the mill juice, and 2.93 per cent over the limed clarification. The glucose was attacked by the excess of lime and .12 per cent converted into organic acids. In the sulphured and limed juice, .22 per cent glucose was attacked. This increase in the destruction of glucose is due to the longer time the excess of lime had to act, as this juice was allowed to cool before sulphuring. The glucose ratio of both clarifications is proportionately reduced. The amount of salts of sulphurous and sulphuric acids remaining in the lime and sulphur clarified juice, No. 5, are small and in proportion to their relative solubilities. The ash of No. 5 is reduced .025 per cent below No. 4, and the lime remaining in solution is .019 per cent, showing that the sulphur aided in removing some of these compounds. The albuminoids and gums are also less in the lime and sulphur clarification, and this is particularly favorable, since it has been noted that the glucose was attacked to a greater extent in this clarification than it was in the clarification with lime alone. The amids are also broken up in each clarification, more so in the lime and sulphur than in the lime clarification. This is also accounted for by the duration of the action of the excess of lime. From these results it will be noticed that on bringing an alkaline clarification back to acidity considerable improvement is made in the character of the juice, and the further action of the excess of lime is checked, which otherwise would continue active during evaporation. By increasing the alkalinity to 3.3 cubic centimeters, another study was made of the effect of sulphur in correcting this larger alkalinity, both in the juice with settlings remaining and in the settled juice. The sulphuring here was carried further (2.7 cubic centimeters acidity) in order to overcome the viscosity and to see its final effect on the gums. Nos. 6, 7 and 8 constitute this experiment. In No. 6 the juice contained 3.3 cubic centimeters excess of lime. In No. 7 the excess of lime was neutralized by sulphurous acid and the sulphuring was continued until the juice was 2.7 cubic centimeters acid In this clarification the precipitate was allowed to remain suspended in the juice. No. 8 was treated as in No. 7, except the juice was settled and the precipitate removed before sulphuring. The purities vary, sulphuring increasing slightly the purity in

the unsettled juice and showing a decrease in the purity of the settled juice. The glucose, while less than in the limed juice, shows a much greater destruction in the settled than in the unsettled juice. Sulphurous acid, both free and combined, is present in larger quantities than heretofore noted. This is to be expected from the acidity of the juice. The amount left in the settled juice is nearly one-half less than that left in the unsettled juices. The lime remaining in solution is practically the same in the limed and in the limed and sulphured juice unsettled, but a very appreciable increase is noted in the settled juice. These variations are due largely to the action of the sulphurous acid on the lime, both combined and mechanically held in the impurities. The ash of both settled and unsettled juices is more than that in the limed juice.

In the experimental runs as shown in Tables XXXIII and XXXIV the juice was sulphured to 5 cc. N-10 acidity and brought back with lime to 0.2 N-10 acidity per 10 cc. of juice. The evaporation and boiling were conducted in the usual way.

Practical exhaustion of the molasses in experiment I was obtained in two boilings. Contrary to what might be expected, a decided difference will be observed on comparing the record of this clarification with those of the alkaline class, especially as regards the increase in the purity of the clarified juice. Not the least evidence of inversion is noticeable; in fact, there is a perceptible falling off in the glucose ratio of the sulphured and clarified juices, due, perhaps, to a slight destruction of the reducing sugar during sulphuring and liming.

Inasmuch as the sulphitation process is the one most commonly practiced in Louisiana, a somewhat fuller account of the changes which take place in the clarification and boiling may be permitted.

The first action of the sulphur dioxide on entering the juice is to cause a coagulation of the albuminoid matter. A flocculent precipitate is formed which carries down not only the albuminoids of the juice, but mechanical impurities such as the fat and wax, particles of fibre and earthy matter from the cane, a considerable amount of gums, etc. The quantity of precipitate which will deposit after sulphuring amounts to from .3 to .4

TABLE XXXIII.

Sulphured to 5 cc. acidity.
Limed to 0.2 cc. acidity.

Striped and Purple Plant Cane.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	13.24	9.62	1.14	1.04	72.66	22.66	0.38	0.10	0.12	0.09	0.102	0.163
Sulphured Juice	13.21	9.76	1.15	.95	73.88	21.51	0 38		0.12	0.07	0.019	0.193
Clarified Juice	13.11	9.89	1.12	.93	75.44	20.73	0.42	0.03	0.19	0.12	0.023	0.176
Syrup	50.00	36.15	4.45	4.11	72.30	23.68	1.66	0.14	0.84		0.119	0.663
First Masse cuite	90.00	69.54	8.57	8.07	77.28	23.92	3.08				0.165	1.200
First Sugar		95.80	2.66				0.72					
First Molasses	80.00	44.02	14.50	12.58	55.03	61.55	5.07		3.09	1.94	0.276	1.847
Second Masse cuite	90.00	48.25	16.38	13.81	53.60	62.58	5.78				0.287	2.248
Second Sugar		84.34	2.79	2.29			3.72					
Second Molasses	80.00	33.68	15.83	16.21	42.10	95.14	7.00				0.265	2.806

TABLE XXXIV.

Run 6. Sulphured to 5 cc. acidity.
Limed to 0.2 cc. acidity.

D. No. 74. Plant Cane.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	14.97	11.97	0.73	0.84	80.00	13.11	0.47	0.13	0.18	0.09	0.097	0.116
Sulphured Juice	15 14	12.32	0.67	0.69	81.37	11.04	0.41		0.12	0.07	0.022	0.109
Clarified Juice	14.97	12.44	0.65	0.62	83.10	10.21	0.48	0.02	0.18	0.12	0.022	0.110
Syrup	50.00	41.02	2.14	2.22	82.04	10.62	1.73	0.08	0.84	0.62	0.177	0.288
First Masse cuite	90.00	76.13	4.04	3.78	84.59	10.26	2.98				0.159	0.696
First Sugar		94.10	2.08				0.91					
First Molasses	80.00	50.35	7.36	7.41	62.97	29.34	6.59				0.265	1.416
Second Masse cuite	90.00	56.85	8.01	9.24	63.18	30.35	6.97				0.294	1.800
Second Sugar		86.07	1.80	2.11			3.52					
Second Molasses	80.00	38.40	11.58	12.63	48.00	63.05	10.24				0.331	2.200

per cent of the weight of the juice. The composition of the air-dried sulphur precipitate for two seasons at the sugar-house of the Experiment Station is shown in the following table:

TABLE XXXV.

(Composition of Sulphur Precipitate.)

	Season of 1903.	Season of 1904.
Moisture	4.07%	4.49%
Fat and wax	32.57%	19.71%
Protein	23.63%	21.75%
Ash and earthy matter	9.48%	20.45%
Crude fibre	8.05%	10.37%
Gums, etc	22.20%	23.23%
	100.00	100.00
Protein insoluble in pepsin (Nuclein bodies)	9.63	.....

The large amount of fat and wax contained in the precipitate is especially noteworthy; in fact, the dried deposits offer a most excellent material for the preparation of cane-wax. By boiling the finely ground precipitate with strong alcohol and filtering hot, the wax will crystallize out almost immediately. By filtering and recrystallizing several of them the wax of melting point 82° C. can be obtained perfectly pure.

If the sulphur precipitate could be removed before liming, the results accomplished by the subsequent clarification would be much more favorable. The removal of the deposits by filtration or sedimentation does not seem feasible, however, in commercial work. When lime is added and the juice heated a part of the suspended precipitate is redissolved. A portion of the gums pass again into solution; the nuclein compounds also appear to undergo a partial disintegration with the formation of xanthin and other nitrogenous bases, with the result that the percentage

of nitrogen in the clarified juice usually exceeds that of the filtered sulphured juice. The lime uniting with the soluble sulphates and phosphates of the juice causes a considerable precipitation of these as insoluble salts of lime, which, with the undissolved matter of the sulphur precipitate constitute a greater part of the filter press cake. A large amount of the added lime, however, is retained in solution as soluble sulphite, so that the percentage of ash in the clarified juice may be even greater than before sulphuring.

The lime, which in the clarified juice and syrup exists in the form of soluble sulphite, undergoes a very rapid oxidation during the subsequent operations of the sugar-house, and in from four to eight weeks is largely changed to the form of sulphate. A large amount of insoluble sulphate of lime crystallizes out in the second masse cuite in the hot room, and being held back mechanically by the sugars in the centrifugals, causes a very marked increase in the ash content of the second sugars, as compared with the firsts. If the second sugars are dried very late a greater part of the insoluble lime sulphate may be removed at this stage, with the result that the second sugar contains a considerably higher percentage of ash than the thirds.

Other changes produced in cane products during the processes of manufacture will be referred to under the composition of cane molasses.

ACID CLARIFICATION WITH SULPHUROUS ACID, LIME, AND PHOSPHORIC ACID.

This method of clarification, which is practiced to a limited extent in Louisiana, consists in liming the sulphured juice to alkalinity and then bringing back to neutrality with phosphoric acid. The method gives usually a very fine clarification, but requires delicate manipulation at the latter stage of the process. If an excess of phosphoric acid is used the sulphite of lime is decomposed during the boiling, with the liberation of sulphur dioxide, which produces an inversion of sucrose.

In the following experiment the juice, after sulphuring to 5cc. N 10 acidity (10cc. juice), was limed to 1cc. alkalinity, and after heating and skimming was treated with phosphoric acid to 0.2cc. acidity:

TABLE XXXVI.

Sulphured to 5 cc. acidity.
Limed to 1 cc. alkalinity.
Phosphoric acid to 0.2 cc. acidity.

Second Year Stubble Cane. D. No. 74.

	Total Solids	Sucrose	Dextrose	Levulose	Purity	Glucose Ratio	Ash	Free Acid	Combined Acid	Gums	Albuminoids	Amids
Raw Juice	16.02	13.64	0.67	0.75	85.14	8.86	0.47	0.11	0.11	0.09	0.080	0.038
Clarified Juice	15.77	13.40	0.63	0.59	84.97	9.10	0.46	0.02	0.18	0.13	0.016	0.042
Syrup	50.00	39.97	2.65	2.62	79.93	13.21	1.81	0.09	0.82	0.47	0.118	0.118
First Masse cuite	90.00	73.76	4.81	4.62	81.95	12.26	3.29				0.185	0.206
First Sugar		92.30	2.34									
First Molasses	80.00	45.74	9.13	8.92	57.18	39.46	6.42				0.322	0.390
Second Masse cuite	90.00	51.44	10.46	10.93	57.16	41.78	7.18				0.332	0.425
Second Sugar		87.15	3.79									
Second Molasses	80.00	34.40	13.38	13.74	43.00	78.83	8.82				0.375	0.718

Superheat Clarifications.

In these clarifications the results from the ordinary method of heating in an open vessel and that of heating under pressure (superheat) in a closed vessel are compared.

Table XXXVII shows the results with lime as the only clarifying agent:

The comparison of the action of lime alone in the ordinary and superheat clarifications, as shown in Table XXXVII, will be interesting.

In analyses No. 1 to No. 6, inclusive, the same degree of acidity is left after clarification and the clarified juices, 2, 3, 5 and 6, are comparable, 2 and 5 being clarified by ordinary method, and 3 and 6 by superheat. It will be noted that in both juices the effect of clarification was the same, the slight variations being about equally distributed and well within the limits of analytical error. In these clarifications the acidity of the juice was not all neutralized by lime, but .55 cubic centimeter acidity was allowed to remain, leaving us a fairly acid juice. Following this was an increase in the quantity of lime until the juice was very slightly alkaline (.1 cubic centimeter).

Comparing the superheat with the ordinary clarification, the same similarity in these results is noted as that occurring in the more acid juices, and again no appreciable difference is recorded. In examining Nos. 13, 14 and 15, where the liming has been carried on to alkalinity, the clarified juice being left .9 cubic centimeter alkaline, there is a considerable increase in the purity of the superheat clarification over both the mill juice and ordinary clarification. The superheat clarification showed 2.64 per cent increase in purity over the mill juice, and the ordinary clarification only .65 per cent increase. While this is quite a favorable increase and the other results are the same, or within very close limits, it must be noted that the glucose is attacked and there is a corresponding increase in gums. Both are slight, yet the gums in the clarified juices are higher than in the mill juice, and more than offset the increase in purity noted in the superheated clarification.

From these results no advantage or disadvantage can be placed to the superheat or the ordinary clarification from the action on the impurities, the results being practically identical

except in alkaline juice, where the advantage is with the superheat clarification, though in this method the injury of the excess of lime would debar it from practice.

SUPERHEATED CLARIFICATIONS WITH LIME AND SULPHURED CANE JUICES.

These tests of the two methods of clarification were made as a continuation of the comparison of the superheat clarification with lime alone as a clarifying agent. Table XXXVIII shows the results:

In Table XXXVIII acid, neutral and alkaline clarifications are given, using nearly the same amount of sulphurous acid in each clarification.

The first comparisons of the clarifications were of a mill juice sulphured to 5.5 cubic centimeters acidity and lime added until the acidity was reduced to .5 cubic centimeter. Comparing the ordinary clarification with the mill juice, there is but slight variation in the solids or sugar content. Of the abuminoids, 74 per cent were removed and the gums show an increase of 21.9 per cent. The superheat clarification, No. 3, has a very slight increase in purity, not enough to justify any material benefit, but the decrease in glucose is, while slight, worthy of notice. The albuminoids remained the same and the gums increased over those present in the ordinary clarification. the increase being 31 per cent over those in the mill juice. In the neutral clarifications and the mill juices, Nos. 4, 5 and 6, the purity and glucose ratio are practically the same and nearly all of the albuminoids are removed in both clarifications. Of the gums 64.8 per cent are removed by ordinary clarification and 70 per cent in the super heat clarification, an advantage to superheating. The alkaline clarifications and mill juice from which they were obtained are given as Nos. 7, 8 and 9. Here increases of 1.11 per cent and 1.70 per cent in purity over that of the mill juice for the ordinary and superheat clarifications, respectively, are noted. The glucose is practically the same in the mill and both clarified juices.

The albuminoids are removed in the same quantity by both clarifications and gums in greater quantity by the ordinary clarifiction. Thirty-two and five-tenths per cent of those present in

TABLE XXXVII.

	Acidity of Juice	Alkalinity of Juice	Brix	Sucrose	Glucose	Solids Not Sugar	Purity Coefficient	Glucose Ratio	Total Proteids	Albuminoids	Amids	Alcoholic Precipitate	Gums, etc.	Ash in Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate
1. Mill juice	1.55	.....	14.61	12.00	1.14	1.47	82.13	9.50	.155	.083	.072	.181	.074	.046	.061
2. Ordinary clarification	.55	.....	14.46	12.00	1.14	1.32	82.98	9.50	.104	.029	.075	.107	.048	.050	.009
3. Superheat clarification heated to 123° C.	.55	.....	14.46	12.00	1.14	1.32	82.98	9.50	.104	.037	.067	.104	.053	.044	.007
4. Mill juice	1.60	....	15.24	12.00	1.78	1.46	78.74	14.83	.165	.065	.105	.148	.047	.049	.052
5. Ordinary clarification	.55	.....	15.24	12.00	1.80	1.44	78.74	15.00	.078	.017	.061	.117	.057	.052	.008
6. Superheat clarification heated to 123° C.	.55	.....	15.24	12.00	1.78	1.46	78.74	14.83	.075	.017	.058	.106	.156	.046	.004
7. Mill juice	1.80	.....	14.85	12.00	1.46	1.39	80.80	12.17	.121	.069	.052	.212	.071	.073	.068
8. Ordinary clarification	....	0.10	14.61	12.00	1.45	1.15	82.13	12.09	.068	.013	.055	.099	.058	.029	.012
9. Superheat clarification heated to 123° C.	.....	0.10	14.62	12.00	1.42	1.20	82.08	11.84	.068	.013	.055	.090	.052	.029	.009
10. Mill juice	1.85	.....	15.00	12.00	1.38	1.62	80.00	11.50	.151	.089	.062	.194	.054	.076	.064
11. Ordinary clarification	....	0.10	15.00	12.00	1.38	1.62	80.00	11.50	.101	.016	.085	.050	.024	.023	.003
12. Superheat clarification heated to 123° C.	.....	0.10	15.00	12.00	1.35	1.65	80.00	11.25	.089	.015	.074	.056	.022	.028	.006
13. Mill juice	2.1	.....	15.00	12.00	1.50	1.50	80.00	12.50	.116	.071	.045	.1471	.066	.023	.058
14. Ordinary clarification	.....	0.90	14.88	12.00	1.32	1.56	80.65	11.00	.061	.010	.051	.128	.074	.019	.035
15. Superheat clarification heated to 123° C.	.....	0.90	14.52	12.00	1.33	1.19	82.64	11.09	.067	.013	.054	.134	.071	.022	.041

TABLE XXXVIII.

	Acidity	Alkalinity	Brix	Sucrose	Glucose	Solids Not Sugar	Purity Coefficient	Glucose Ratio	Gums and Acids	Total Proteids	Albuminoids	Amids	Alcoholic Precipitate	Gums, etc., in Alcoholic Precipitate	Ash in Alcoholic Precipitate	Albuminoids in Alcoholic Precipitate
1. Mill juice	2.40	....	15.85	12.00	2.40	1.45	75.71	20.00	....	.111	.062	.049	.212	.128	.050	.034
2. Ordinary clarification sulphured to 5.5cc acidity and limed to .5 cc. acidity	0.50	.....	15.83	12.00	2.43	1.40	75.81	20.25	.....	.062	.016	.046	.245	.156	.071	.018
3. Sulphured clarification of No. 2 heated to 130° C.	0.50	.....	15.79	12.00	2.33	1.46	76.00	19.42	...	.059	.016	.043	.276	.169	.090	.017
4. Mill juice	1.55	.....	14.32	12.00	1.51	0.81	83.79	12.58	.....	.104	.052	.052	.176	.053	.060	.053
5. Ordinary clarification sulphured to 6cc. acidity and limed to 0.1 cc. alkalinity	.....	0.10	14.31	12.00	1.53	0.78	83.86	12.75	.....	.048	.007	.041	.062	.043	.016	.003
6. Superheat clarification of No. 5	.....	0.10	14.23	12.00	1.50	0.73	84.33	12.50	.....	.045	.003	.042	.052	.042	.010	.0005
7. Mill juice	2.20	.....	14.85	12.00	1.69	1.16	80.81	14.08	....	.133	.063	.070	.234	.111	.082	.041
8. Ordinary clarification sulphured to 5.2cc acidity and limed to 1.0 cc. alkalinity	.....	1.00	14.65	12.00	1.68	0.97	81.92	14.00	.....	.078	.013	.065	.158	.057	.090	.011
9. Superheat clarification of No. 8 heated to 130° C	.....	1.00	14.54	12.00	1.65	0.89	82.51	13.75	.....	.080	.014	.066	.179	.079	.087	.013

the mill juice are removed by ordinary clarification, while 23.5 per cent are removed by the superheat clarification.

Here the ordinary clarification gives better results than the superheat.

In comparing the above we find the greatest variation in the amount of gums left in the clarified juices, the ordinary method of clarification giving better results in acid and alkaline clarifications and the superheat having the advantage in neutral clarification. None of these advantages are great, and it may be said that with sulphur and lime used in clarifying there is very little difference between the superheat and the ordinary clarifications on mill juice.

Reviewing the action of sulphur and lime on clarified juices, we note that the greatest efficiency was obtained when sulphur was used in large quantities and then neutralized with lime, sulphuring to 10 cubic centimeters acidity and liming to neutrality giving the best clarification and removing a considerably greater percentage of gums than any other combination of lime and sulphur. The use of lime followed by sulphur shows in no instance any advantage over sulphuring first, and in some clarifications a distinct disadvantage.

Neutral clarifications give the best results, leaving less impurities of all characters than acid or alkaline clarifications. Albuminoids are largely removed by all clarifications, but the largest percentage is removed by the neutral. Amids are not removed by any process, but are increased by alkaline clarifications, though in some instances they are decomposed with a liberation of ammonia and formation of organic acids.

PHOSPHORIC ACID AND LIME CLARIFICATIONS.

Phosphoric acid is used in clarification to remove the excess of lime added to the juice. It unites with lime, forming phosphate of lime, which is practically insoluble in the juice.

The following table gives the superheat clarification, using phosphoric acid and lime as clarifying agents:

TABLE XXXIX.

	Acidity	Alkalinity	Brix	Sucrose	Glucose	Solids Not Sugar	Purity Coefficient	Glucose Ratio	Total Proteids	Albuminoids	Amids	Alcoholic Precipitate
1. Mill juice	1.50	.....	15.15	12.00	.....	.....	79.21	.....	.103	.052	0.51	.173
2. Ordinary clarification limed to 1.5 cc. alkalinity and Phosphoric Acid added to 0.1 cc. acidity	0.10	...	15.00	12.00	....	.....	80.00	.....	.079	.013	0.66	.014
	0.10	.....	14.75	12.00	.....	.....	81.35	.....	.065	.013	.052	.029
3. Superheat clarification of No. 2 heated to 130° C	1.75	.....	14.29	12.00	1.40	0.89	83.98	11.67	.092	.050	.042	.148
4. Mill juice												
5. Ordinary clarification limed to 2 cc. alkalinity and Phosphoric Acid added to 0.5 cc. acidity	0.50	.....	13.97	12.00	1.40	0.57	85.89	11.67	.053	.009	.044	.067
	.050	.....	14.02	12.00	1.45	0.57	85.59	12.08	.048	.006	.042	.079
6. Superheat clarification of No. 5												

On comparing the results in the above table, it is first to be regretted that, owing to an accident in the laboratory, they are only partially complete. However, there are enough data to judge the merits of the clarification.

Having seen the disadvantages of heavy liming, followed by either sulphurous or phosphoric acid, and having noted the danger from leaving juices alkaline, it was thought best to make these clarifications slightly acid, or, in the cases of Nos. 1, 2 and 3, nearly neutral.

The first clarifications, Nos. 2 and 3, were limed to 1.5 cubic centimeters alkalinity, then, without settling, phosphoric acid was added to slight alkalinity (.1 cubic centimeter). Here an increase in purity over the mill juice is noted both by the superheat and ordinary clarifications, the ordinary causing an increase of .76 per cent and the superheat 1.14 per cent. The albuminoids removed are the same, 75 per cent of those present in the mill juice in each clarification. The gums were not estimated, but the alcohol precipitate which contains them shows only a very small quantity remaining in the ordinary clarification, while in the superheat clarification the alcohol precipitate is double the quantity of the ordinary, and a proportionate increase in gums in this clarification over the ordinary clarification is indicated.

Continuing further these clarifications, increasing the quantity of lime to 2 cubic centimeters alkalinity and adding phosphoric acid to .5 cubic centimeter acidity, there is noted an increase in purity in both ordinary and superheat clarifications, the ordinary clarification showing the greater increase. The glucose is not changed in the ordinary clarification and shows a slight increase in the superheat clarification. The albuminoids are largely removed by both procedures, 82 per cent being removed by ordinary clarification and 88 per cent by superheat. The gums, etc., were not directly determined. The alcohol precipitate shows evidences of their removal in considerable quantity, though, as was to be expected, a less amount was removed by the strongly acid clarification than by the nearly neutral ones. The superheat here again gave evidence that more gums remain in the clarified juice here than in the ordinary clarification.

The increase in glucose ratio and decrease in purity between the raw juice and syrup show that a very noticeable inversion took place in the early stages of the process.

We have already called attention to the fact that the use of phosphoric acid in clarification occasionally gives trouble by rendering the molasses turbid through formation of insoluble lime phosphate. This is also the case when a dilute solution of phosphoric acid is used for washing sugars in the centrifugals (a questionable practice at the best) and the washings are allowed to flow into the molasses. A number of cases have been reported where open kettle molasses depreciated in market value as a result of the turbidity resulting from this cause.

As a general summary of the foregoing experiments upon alkaline and acid clarifications, we may say that for Louisiana conditions, where the juices usually contain a high percentage of reducing sugars as compared with tropical countries, a carefully conducted sulphitation gives the most satisfactory results from the point of economy, as well as from the favorable outturn of sugar. It is needless to add that in this, as in all other methods of clarification, satisfactory results can be secured only when the process is subjected to a most rigid chemical control.

OTHER EXPERIMENTS IN CLARIFICATION.

From time to time notices appear of startling results obtained by some new process of clarification. The announcement, however, is usually soon forgotten, the experiment joining the silent majority of those pronounced non-feasible. In one of the foreign sugar journals some years ago appeared a list of nearly three hundred chemicals which had been tried or proposed for clarifying saccharine juices, but it is doubtful if the following method, which the Sugar Experiment Station was requested to investigate some time ago, is in this catalog.

The inventor proposed to make use of the rare mineral, *monazite*, which occurs in Brazil, and also to some extent in North Carolina. This mineral, which consists of a beautiful gold-colored sand, contains a number of rare metals, the oxides of which are largely used at present in the preparation of the well-known Welsbach mantles for gas burners. The clarifying agent in this new method consists of the gelatinous hydrates of

these same rare earths. Very extravagant claims were made for this process. We quote as follows: "The gelatinous oxides of monazite prevent inversion and alcoholic fermentations, precipitate pigments and albuminoid matter, and consequently clarify and bleach the juice." The inventor then states: "At first one might suppose that this process would not be practical commercially on account of the cost of the clarifying agent, but it is not so, for the gelatinous oxides of the monazite are recovered after the clarification, calcined to destroy organic impurities, then dissolved in nitric acid and reprecipitated by ammonia for further use." The complications of recovery would alone impede the introduction of this process, but aside from this, experiments showed that the hydrates from monazite possessed no advantages in clarifying beyond that of any other gelatinous oxide. Hydrated alumina was found to answer equally well, if not better, and alumina has long been discarded as a clarifying agent in the sugar-house.

Another method of clarification, which the Experiment Station examined, seemed to possess, at first sight, greater possibilities of commercial success than the one just described. This was the electrical process, of which we hear so much from time to time. In this process a strong electric current was passed through the juice between electrodes composed of an alloy of aluminum and magnesium. Considerable heat was developed, the juice was bleached, and a flocculent precipitate thrown down. An analysis of the juice before and after clarification gave the following results:

	Before. Clarification.	After Clarification.
Brix	15.55	15.39
Sucrose	12.20	12.30
Glucose	1.98	1.79
Ash	0.31	0.35
Freed acid cc. N-10 alkali to 100gms...	10cc.	4cc.
Coefficient of purity	78.1	79.9

The analysis shows no inversion of sucrose and a decided gain in purity. The acidity is reduced over one-half, but the marked increase in ash shows that this is largely the result of the action of the acids of the juice upon the electrodes. The

clarifying effects of the process seem to be due partly to the influence of the heat developed, partly to the action of the dissolved alumina, and partly to the action of the gases generated during the electrolysis.

The great expense connected with this method of clarification will prevent its general introduction. The installation and renewal of electrodes is very costly and such an outlay of electrical energy is required to accomplish satisfactory results that the undertaking cannot be made profitable.

VII. SCHEMATIC TABULATION OF YIELDS AND COMPOSITIONS OF SUGAR-HOUSE PRODUCTS.

As a general summary of the experiments previously described, the yields and compositions of the different products obtained from various qualities of cane juice have been condensed into tabular form. The tables which are given below do not represent any particular experiment, but are made up from the average results of many years' work in the sugar-house and laboratory. The figures showing the yield of products are obtainable only under the best of sugar-house conditions when there are no losses from inversion or entrainment.

At the beginning of the sugar season in Louisiana the juices are of relatively low sucrose content and of inferior purity, and two boilings are usually sufficient to obtain all the available sugar. Table XL gives the yields and compositions of the various products obtained with a low grade juice of this description. Table XLI gives the results obtained later in the season with an average juice, and Table XLII the yields and compositions of products from a high grade juice toward the close of grinding.

All calculations were performed upon the basis of 1,000,000 pounds of cane and an extraction of 75 per cent. The compositions of syrups, molasses and masse cuites were all made upon a percentage of total solids of 50, 80 and 90, respectively, these figures being the general average, though individual instances may show considerable variation from these percentages.

TABLE XL.

(Yields and Compositions of Sugar House Products from a Poor Juice.)

	Pounds	Total Solids	Sucrose	Dextrose	Levulose	Ash	Albuminoids	Amids, etc.	Acids, Gums, etc.
Cane	1,000,000								
Raw Juice	750,000	13.50	10.00	1.20	0.80	0.45	0.10	0.15	0.80
Sulphured Juice	750,000	13.30	10.00	1.20	0.80	0.40	0.02	0.15	0.73
Clarified Juice	750,000	13.20	10.00	1.20	0.80	0.45	0.03	0.15	0.57
Syrup	198,000	50.00	37.90	4.40	3.20	1.70	0.10	0.58	2.12
First Masse cuite	110,000	90.00	68.20	7.00	6.60	3.00	0.15	1.05	4.00
First Sugar	48,000	98.75	96.00	0.80	0.70	0.80	0.01	0.04	0.40
First Molasses	64,500	80.00	44.80	11.40	10.80	4.50	0.22	1.78	6.50
Second Masse cuite	57,330	90.00	50.40	12.50	12.50	5.00	0.23	1.97	7.40
Second Sugar	20,000	94.00	80.00	3.90	4.10	3.00	0.06	0.64	2.30
Second Molasses	41,000	80.00	31.40	15.00	16.00	5.60	0.30	2.40	9.30

TABLE XLI.

(Yields and Compositions of Sugar House Products from an Average Juice.)

	Pounds	Total Solids	Sucrose	Dextrose	Levulose	Ash	Albuminoids	Amids, etc.	Acids, Gums, etc.
Cane	1,000,000								
Raw Juice	750,000	15.00	12.00	1.00	0.70	0.45	0.10	0.10	0.65
Sulphured Juice	750,000	14.80	12.00	1.00	0.70	0.40	0.02	0.10	0.58
Clarified Juice	750,000	14.70	12.00	1.00	0.70	0.45	0.03	0.10	0.42
Syrup	220,588	50.00	40.80	3.20	2.58	1.50	0.09	0.33	1.50
First Masse cuite	122,550	90.00	73.44	5.70	4.70	2.70	0.13	0.63	2.70
First Sugar	54,000	98.75	96.00	0.80	0.70	0.80	0.01	0.05	0.39
First Molasses	71,213	80.00	53.60	8.76	8.00	4.00	0.20	0.94	4.50
Second Masse cuite	63,300	90.00	60.30	9.43	9.43	4.50	0.21	1.08	5.05
Second Sugar	24,000	95.50	85.00	2.90	3.10	2.50	0.06	0.34	1.60
Second Molasses	42,563	80.00	41.70	12.20	12.50	5.35	0.25	1.50	6.50
Third Masse cuite	37,833	90.00	46.90	13.30	14.50	6.00	0.26	1.70	7.34
Third Sugar	10,000	94.00	80.00	3 80	4.20	3.50	0.07	0.43	2.00
Third Molasses	30,750	80.00	31.70	15.00	16.50	6.30	0.38	2.00	8.20

TABLE XLII.

(Yields and Compositions of Sugar House Products from a Good Juice.)

	Pounds	Total Solids	Sucrose	Dextrose	Lævulose	Ash	Albuminoids	Amids, etc.	Acids, Gums, etc.
Cane	1,000,000								
Raw Juice	750,000	15.50	13.50	0.60	0.40	0.40	0.10	0.15	0.35
Sulphured Juice	750,000	15.35	13.50	0.60	0.40	0.38	0.02	0.15	0.30
Clarified Juice	750,000	15.30	13.50	0.60	0.40	0.42	0.03	0.15	0.20
Syrup	229,500	50.00	44.10	1.80	1.50	1.37	0.08	0.51	0.64
First Masse cuite	127,500	90.00	79.40	3.10	2.80	2.50	0.12	0.88	1.20
First Sugar	72,000	98.75	96.00	0.80	0.70	0.80	0.02	0.13	0.30
First Molasses	54,563	80.00	58.90	6.00	5.80	4.70	0.25	1.85	2.50
Second Masse cuite	48,500	90.00	66.20	6.65	6.65	5.30	0.26	2.10	2.84
Second Sugar	25,000	96.50	90.00	1.40	1.60	2.50	0.05	0.40	0.55
Second Molasses	24,269	80.00	39.70	11.40	12.00	8.00	0.40	3.50	5.00
Third Masse cuite	21,572	90.00	44.60	12.30	14.00	9.00	0.41	3.69	6.00
Third Sugar	5,000	95.75	85.00	2.80	3.20	3.50	0.06	0.49	0.70
Third Molasses	18,284	80.00	29.40	13.60	15.80	9.80	0.43	3.97	7.00

The results shown in Table XL (two boiling) with a poor juice of low purity indicate a yield of 136 pounds (6.8%) commercial sugars, or 124 pounds (6.2%) pure sucrose and 82 pounds residual molasses (about 7 U. S. gallons) per ton of cane.

Table XLI (three boilings) with an average juice of 80 purity shows a yield of 176 pounds (8.8%) commercial sugars, or 160 pounds (8%) pure sucrose and 61.5 pounds residual molasses (about 5 U. S. gallons) per ton of cane.

Table XLII (three boilings) with a superior juice of nearly 90 purity, shows a yield of 204 pounds (10.2%) commercial sugars, or 192 pounds (9.6%) pure sucrose and 36.5 pounds residual molasses (about 3 U. S. gallons) per ton of cane.

VIII. THE COMPOSITION OF LOUISIANA MOLASSES.

The composition of the first, second and third molasses has been given in many of the preceding tables. A somewhat closer examination of several constituents will be of value, particularly as regards ash and nitrogenous bodies.

The composition of the ash of residual molasses from several sugar-houses in Louisiana is given in Table XLIII.

TABLE XLIII.

(Composition of Ash from Different Molasses.)

		I	II	III	IV
		Mill Sulphitation	Diffusion Sulphitation	Open Kettle	Carbonatation
Potash	K_2O	49.48%	52.20%	51.48%	50.16%
Soda	Na_2O	0.89	0.80	1.11	0.32
Lime	CaO	6.47	6.78	6.58	8.53
Magnesia	MgO	4.29	3.09	3.99	2.66
Iron Oxide	Fe_2O_3	0.35	0.33	0.15	0.47
Alumina	Al_2O_3	0.30	0.22	0.13	0.30
Silica	SiO_2	4.12	4.59	2.83	4.10
Phosphoric Acid	P_2O_5	3.71	3.80	2.12	0.91
Sulphuric Acid	SO_3	10.79	6.72	10.94	11.18
Carbonic Acid	CO_2	7.49	11.19	13.06	15.78
Chlorine	Cl	14.00	11.95	9.10	4.59
Total		101.89	101.67	101.49	99.00
Deduct O = Cl		3.16	2.70	2.05	1.04
		98.73	98.97	99.44	97.96
Undetermined (Carbon, etc.)		1.27	1.03	0.56	2.04
Alkalinity (cc. $\frac{N}{10}$ per gr. ash)		80cc	93cc	95cc	109cc

Comparison of the above analyses with those of the ash of cane juices (Table XXII) shows several very striking differences. There is only about one-half the quantity of sulphuric acid in the molasses ash, notwithstanding the heavy sulphuring which some of the juices received. This, of course, is due to the removal of the sulphuric acid as calcium sulphate; the same holds true of phosphoric acid. We note also an increase in the amount of lime in the molasses ash, with a corresponding increase in carbonic acid and alkalinity and a decrease in iron, alumina and silica. The variations in chlorine content of the four ashes are especially noteworthy, and we have here a good illustration of the influence of local conditions upon the composition of cane products. Molasses I was produced upon a plantation near the Gulf, where the cane fields are occasionally flooded with salt water; molasses IV, on the other hand, was produced on a plantation in the interior of Louisiana, over a hundred miles from the coast. The excess of chlorides in Lower Coast molasses such as I is frequently perceptible to the taste, and this, of course, affects its sale for household purposes.

The distribution of the nitrogen of residual cane molasses among the different bodies shows, as might be supposed, very noticeable differences from what exists in the raw juice.

TABLE XLIV.

(Distribution of nitrogen in sugar cane molasses.)

	Percentage in Molasses.	Percentage of Total Nitrogen.
Nitrogen in albumoses and peptones	0.0153%	3.28%
Nitrogen in amido-acids	0.1774%	38.00%
Nitrogen in amido-acid amids	0.0672%	14.38%
Nitrogen in ammonia	0.0147%	3.15%
Nitrogen in nitrates	0.0370	7.92
Nitrogen in nitrogenous bases (xanthin, etc.)	0.1113%	23.83%
Nitrogen in other forms	0.0441%	9.44%
Total	0.4670	100.00

A comparison of Tables XLIV and XIII shows very marked differences. The large amount of nitrogen in the molasses in the form of xanthin bodies is especially noticeable, those constituents being almost completely absent in the juice. The xanthin and other nitrogenous bases in the molasses are no doubt largely formed by a breaking up of the nucleo-proteids during clarification and by the disintegration of the soluble albumoses during the whole process of manufacture.

Among other ingredients which accumulate in the final molasses in large amounts are the organic acids (principally as lime and potash salts) and the gums. These are derived partly as such from the original juice and partly from the decomposition of the sugars, amids and other organic ingredients during manufacture. Investigations of these constituents are at present being carried out, and we hope in the near future to give a fuller account of their nature and characteristics.

REWORKING OF MOLASSES WITH FIRST PRODUCTS.

In the foregoing descriptions of sugar-house operations, the sugars were obtained from the juice in two or three different operations. A few plantations in Louisiana have made a practice of obtaining all their sugars in one operation without the use of a hot room. In this process the molasses from the centrifugals is either run back into the mill juice before sulphuring and reworked or is taken up into the pan and reboiled after grain has been formed from a fresh lot of syrup. This process is kept up for three or four rounds when the accumulation of impurities becomes so great that its further continuation is undesirable. The molasses is then withdrawn from circulation either wholly or in part, and the process continued as before.

While the above process or some of its numerous modifications may effect a considerable economy of time and space in the sugar-house, it is never possible to obtain the exhaustion attainable by the old hot room method. The following series of analyses by Chiquelin of molasses obtained from four consecutive strikes by a process of the above description and of the final molasses as withdrawn from circulation, will be of interest:

TABLE XLV.

	No.	Total Solids	Sucrose	Glucose	Solids Not Sugar	Purity	Glucose Ratio
First Strike	1	78.09	52.00	14.70	11.39	66.58	28.27
	2	80.00	48.50	16.50	14.99	60.64	34.01
	3	77.77	47.45	16.02	14.30	61.06	33.76
	4	78.25	47.99	15.15	15.11	61.32	31.57
	Average..	78.53	48.99	15.59	13.95	61.90	31.90
Second Strike	5	74.86	45.73	18.87	10.20	61.07	41.26
	6	72.54	40.87	14.70	15.97	57.12	33.52
	7	74.64	46.03	15.38	13.23	61.67	33.41
	8	71.22	39.52	14.28	17.42	55.49	36.13
	Average..	73.06	43.04	15.81	14.21	58.84	38.58
Third Strike	9	81.16	43.05	17.58	20.53	53.04	40.83
	10	80.77	42.60	16.66	21.51	52.74	39.10
	11	79.00	42.08	16.66	20.26	53.26	39.58
	12	79.02	42.26	16.13	20.63	53.48	38.16
	Average..	79.99	42.50	16.76	20.73	53.13	39.42
Fourth Strike	13	80.55	40.65	14.81	25.09	50.34	36.43
	14	83.71	43.55	16.66	23.50	52.02	38.25
	15	80.57	39.47	14.81	26.29	48.98	37.52
	16	81.24	39.80	14.81	26.63	48.99	37.21
	Average..	81.52	40.87	15.27	25.38	50.08	37.35
Final Molasses ...		80.87	39.76	18.34	22.77	49.16	46.12

The results show a continued accumulation of impurities in the molasses after each round, especially at the third and fourth strikes. The mixing of the molasses, juices and syrups of ever-varying composition and purity of course complicates the control of such a method of manufacture. The final molasses by the above process when withdrawn has a sucrose content and purity of ordinary hot room second molases, but the presence of excessive amounts of gummy decomposition products rendered further working of the residue inadvisable.

IX. EFFECTS OF FERMENTATION UPON THE COMPOSITION OF SUGAR CANE PRODUCTS.

Before concluding this bulletin upon the composition of the sugar cane and its products, a brief account of some of the abnormal constituents, which are sometimes formed by the activity of yeasts, moulds and bacteria, will be of value.

The number of micro-organisms which produce decomposition of cane products is almost unlimited, and the chemical changes which develop, especially when several fermentations take place simultaneously, are necessarily very complex. In the brief space at our command we can only take up a few of the typical and more common fermentations.

The most common fermentation which the raw juice of the cane undergoes in Louisiana is not the alcoholic, as might be supposed, but a fermentation designated variously as the viscous, mucilaginous or mannitic. This fermentation is anaerobic in character; a most powerful reducing action in consequence takes place, by virtue of which the juice is rapidly bleached. The liquid becomes thick and ropy, and if the culture be pure the juice will finally set to a perfectly solid jelly. Various organisms may produce this type of fermentation, but the best known member of this class of bacteria is the *Leuconostoc* or *Streptococcus mesenteroides,* the so-called "frog spawn" of the beet sugar manufacturer. This fermentation was one of the first to attract the attention of investigators and the study of its products of decomposition constitutes an interesting chapter in the subject of biochemistry.

Vanquelin in 1822 caused four bottles of cane juice to be sent from Martinique to France. The samples arrived, however, in a very bad condition, the juice having changed to a thick mucilage. Vanquelin therefore busied himself with a study of the gummy matter into which the sugar had been changed, but the methods of organic analysis at that time were in their infancy, and no definite knowledge of the gum seems to have been gained. Peligot, Kircher, Brüning and many others also occupied themselves with the problem, studying the gummy fermentation products of both cane and beet juices. Durin* regarded the constituent of this gum as cellulose and felt so certain of his ground that he took out a patent—a curiosity of its kind—for the "conversion of crystalline sugar into cellulose and for any use which such cellulose can find technically." It was Scheibler who first established the real nature of the product; he proved the gum to be a body very similar to dextrin and named it dextran. Scheibler himself, however, fell into an error, for he re-

*"De la fermentation celluloisque du sucre de canne". Comptes Rendus. 83.128. 1876.

†Zeits. des verein d. deutschen Zucker-Industrie. 1869. p. 472:1874. p. 309:1875 p. 112.

garded the gum not as a fermentation product, but as a substance occurring naturally in the plasms of the beet-cells.

Dextran was prepared from samples of clarified cane-juice, which had undergone the viscous fermentation, by precipitating with 95% alcohol. The gum was filtered off and repeatedly purified by dissolving in dilute sodium hydrate, filtering and precipitating with alcohol acidified with hydrochloric acid. After washing with alcohol and ether, the gum was dried first at 60° and then, after pulverizing, at 100°, and finally at 130°. The product thus obtained was perfectly white and contained 1.65% of ash, mostly Na Cl. The following analysis, calculated to ash free substance, was obtained:

	Found.	Theoretical for $(C_6H_{10}O_5)n$	Theoretical for $3(C_6H_{10}O_5), H_2O$.
Hydrogen	6.54%	6.22	6.40
Carbon	42.50	44.42	42.83

Other analysts have reported for dextran 41.45-43.61% carbon. The formula usually assigned to dextran is $(C_6H_{10}O_5)n$, the same as that of cellulose. The writer, however, is inclined to the belief that dextran is a hydrated product of variable composition.

A sample of dextran weighing .3848 grm. was dissolved to 50 cc., a drop of ammonia being added to secure freedom from opalescence. The solution gave a polariscopic reading of +4.47 Ventzke in the 100 mm. tube, from which the specific rotation $\left[\begin{smallmatrix} a \\ \end{smallmatrix}\right]^{20°}_{D} = \frac{.3468 \times 4.47 \times 50}{.3848} = +201.8$. The results recorded in the literature for the specific rotation of dextran vary from +195 to +230.

The presence of dextran in sugar-cane products may introduce an error into the analytical work. It happens occasionally in Louisiana that the sugar cane is damaged by a splitting freeze; on the occurrence of warm weather a fermentation sets in, with the formation of considerable dextran within the cane. In analyzing juices from such canes the inexperienced chemist is often puzzled because his juices polarize well, yet give him poor returns in the sugar-house. The following analyses of badly fermented cane-juices will show the influence of dextran upon the polarization:

TABLE XLVI.

No.	Degrees Brix.	Polarization.	Sucrose.	Reducing Sugars.	Dextran.	Apparent Purity.
1...	7.8	+18.0	0.0%	0.15%	5.90%	232
2...	4.8	+10.4	0.0	trace	3.35	216

The occurrence of dextran in cane syrups and molasses might lead the food chemist to suspect an adulteration of these products with commercial glucose, when in reality no such adulterant was present.

The viscous fermentation, as was stated, exerts a powerful reducing action upon the cane juice, and as a consequence of this reduction various deoxidation products are formed. The most common of these is mannite, which was very early recognized among the products of this fermentation, and for this reason the name mannitic fermentation was sometimes applied. It was at first supposed that the mannite was the product of a special organism, but this is a mistake, for mannite may be formed in any fermentation of sugar where a reducing action takes place. The quantity of mannite in fermented juices will vary; juices which showed over 2% mannite were found on subsequent analysis to be nearly deficient in the same, owing to the fact that other fermentations had set in, whereby the mannite was destroyed.

Among the products of the different anaerobic fermentations to which cane juice is subject are various gaseous bodies. In cane juices clarified by the sulphitation process, especially such as have been afterwards treated with phosphoric acid, fermentation occasionally sets in and large quantities of hydrogen sulphide are evolved. The odor of this gas is usually very noticeable around fermenting press cakes. In other cases hydrogen is given off and serious accidents have been reported through the explosion of this gas, generated from juice or syrup that had been left standing in vacuum pans or effects. When fermentation of the juice and bagasse together takes place, as may occur in a diffusion battery, the cellulose of the cane fibre undergoes a decomposition, and the gas given off contains methane as well as hydrogen. Explosions of diffusion cells in sugar beet factories from this cause have been reported not in-

frequently. The fermentation which takes place in bagasse piles is usually of the butyric order, as may be recognized by the peculiar rancid odor which is given off.

Reference was just made to Durin's patent for obtaining cellulose from sugar. Notwithstanding the fact that Scheibler proved Durin's cellulose to be an entirely different body, cellulose may be formed from sugar in large amounts by the activity of bacteria. A fermentation of this kind was reported in the Louisiana Sugar Planter (Vol. 34, p. 238) by the writer several years ago, and more recent investigations show that this fermentation is one of very general occurrence in Louisiana.

This fermentation, unlike the viscous, is aerobic. Large gelatinous lumps of leather-like toughness are formed in the juice. These lumps, which sometimes weigh several pounds, are stratified in appearance and are, as a matter of fact, made up of an infinite number of closely compacted membranes. The substance of these membranes on boiling with alkali does not pass into solution, as is the case with dextran, but shrivels up into a dense white body, which gives all the reactions of cellulose, yielding a blue coloration with zinc chloride and iodine and being 99 per cent soluble in cupro-ammonium. The percentage composition of the purified substance precipitated from cupro-ammonium agreed with that for cellulose.

	Found.	Theoretical for $(C_6H_{10}O_5)n$
Hydrogen	6.28 per cent.	6.22 per cent.
Carbon	43.87 per cent.	44.42 per cent.

The amount of dried membrane formed by this fermentation in a cane juice was about 13% and the amount of cellulose about 7% of the total sugar fermented. When examined under the microscope the membranous tissue is seen to consist of interwoven chains of bacteria imbedded in the capsular matter, which constitutes the real substance of the membrane. Intermingled with these bacterial chains a great many yeast cells are usually visible, and it may be that we have here a case of symbiosis, such as occurs with the ginger beer ferment, which was formerly employed quite extensively in the Southern States for making molasses beer.

As constituents of the deposits and scums which always form in fermenting juices, syrups and molasses, we have a number of substances which, like dextran and cellulose, are to be regarded as of assimilative rather than of fermentative origin. Such, for example, is mannan; the mixed sediment of yeast cells, mycelia, etc., found in decomposed juices and syrups, always contains this body.

Another common ingredient of these fermentation products is the nitrogenous body chitine. We have found this substance to be a very important constituent of the scums* which form every year upon the surface of molasses left over in the hot room. These scums, upon washing out the adherent molasses, constitute a brownish-colored pulpy mass, a sample of which air-dried gave the following analysis:

Moisture	10.00%
Chitine	11.30%
Protein	31.62%
Fat	27.50%
Ash	5.58%
Undetermined (N. free)	14.00%

After extracting the dried material with successive portions of ether, boiling soda, hot water and alcohol, about 15% of insoluble residue was obtained, which analysis showed to be 90% chitine. This residue on boiling with concentrated hydrochloric acid gave a dark-colored solution, which on evaporation yielded beautiful glistening crystals of glucosamine hydro-chloride, $C_6H_{11}(NH_2)O_5HCl$. These crystals were easily soluble in water and gave in the polariscope a specific rotation of +70.45, a little higher than that of sucrose.

The large amount of fat in the molasses scums (27.50%) is noteworthy, and what is more remarkable, the composition of this fat, as is shown from its physical and chemical constants, agrees very closely with that of butter fat.

*These scums are produced by a fungus which has since been identified by Mrs. F. W. Patterson of the Bureau of Plant Industry, U. S. Department of Agriculture, as belonging to the genus citromyces.

	Fat from Scums.	Butter Fat.
Saponification number	223.1	228.5
Iodine absorption number	28.17	33.35
Reichert-Meissl number	30.36	28.3
Melting point	35°	33.2°
Melting point insol. acids	41°	41.7°
Iodine number insol. acids	30.53	29.5

This is the first instance so far as can be found of any other fat, either vegetable or animal, showing such a similarity, as regards the above constants, to butter fat. In certain respects, however, the fat differs from fresh butter fat.

	Fat from Scums.	Butter Fat.
Acid number	85.2	0.50
Ether number	137.9	228.0
Mean mol. wt. soluble acids	129.7	98.1
Mean mol. wt. insoluble acids	283.2	261.0

The distinguishing characteristic of the fat from the scums is the high degree of acidity and the greater preponderance of soluble acids as caproic and caprylic. The high acid number is undoubtedly the result of hydrolysis through a fat splitting enzyme.

It will be impossible at present to take up other interesting fermentations of sugar-cane products, such as that produced by *oiedum lactis*, by the various varieties of *mucor*, *aspergillus*, and *penicillium*, and by the various kinds of *mycoderms*, or to discuss the character of the products formed by these various organisms. There is one fermentation product, however, which we would like to mention before concluding, as it seems to be of quite common occurrence, although not generally recognized. The substance in question is dimethylketol or acetyl-methyl-carbinol. $CH_3CO\text{-}CHOH\text{-}CH_3$. The compound was first made synthetically by Pechman*; it was discovered later by Grimbert† among the products produced by the fermentation of dextrose, dextrin and mannite by *Bacillus tartricus*, and by Browne‡ among the fermentation products in cider vinegar. Pastereau§ has also recently shown this body to be a common constituent of commer-

*Ber. d. Chem. Ges: 21. 2754. 22. 2214.
†Comptes Rendus 132. 706.
‡Jour. Amer. Chem. Soc. 25, p. 31.
§Jour. Pharm Chem. 1905. 21. 593.

cial vinegar. We have again found the same substance in a fermented cane syrup, and it is our belief that this compound is always produced in small amounts whenever the alcoholic fermentation is arrested through the development of oxidizing or acid producing bacteria.

It is a mistake to suppose that the fermentation of sugar-cane products is limited entirely to such dilute media as juices and syrups. Molasses is also very susceptible to fermentation, and even raw sugars during transport or in storage may undergo a gradual deterioration through the activity of yeasts and bacteria. The fermentation of such a thick menstruum as molasses, however, is confined entirely to the surface, which, through the attraction of hygroscopic moisture, becomes dilute enough to favor micro-organic growth. The same is true of raw sugars; the film of molasses coating the crystals undergoes a gradual fermentation, with the result that the underlying sucrose is slowly dissolved and inverted.

The deterioration of molasses and sugar during storage is a problem of great importance to the sugar industry, and the following instance may perhaps have an interest for those who are sometimes disposed to hold their sugars for an increase in market value.

In Apirl, 1904, a shipment of Cuban sugars from different plantations was received in New Orleans. The sugars were of the ordinary 96 test type and showed no particular abnormalities. Polarizations were made at the Sugar Experiment Station after unloading and again of the same samples the succeeding winter. The results are given in Table XLVII.

TABLE XLVII.

(Showing deterioration of sugars on standing.)

Plantation—	Polarization— April, 1904.	Polarization— January, 1905.	Decrease.
Cabo	96.50	95.60	0.90
Fajardo	96.05	95.00	1.05
Mercedita Central	95.50	93.20	2.30
Toledo	94.20	91.70	2.50
Ramon	97.15	94.60	2.55
Caridad	93.95	91.10	2.85
Providencia	94.70	91.20	3.50
Mercedita	95.00	91.20	3.80
Nombre de Dios	95.90	91.50	4.40
Avenhoff	96.80	90.70	6.10
Lucia	96.20	89.00	7.20
Average	95.63	92.25	3.38

At the time of the second polarization the sugars had all perceptibly darkened and had acquired a very marked acid reaction. Bacteriological tests showed the number of organisms to vary from 64 to 512 per gram of sugar. The bacteria were largely aerobic in character, and, finding a very suitable medium for growth in the moist film of molasses coating the sugar crystals, produced an inversion which resulted in a decrease in polarization of from 0.1 to 0.8 per cent per month. In the summer the deterioration was, no doubt, much greater than this general average. The thorough drying of commercial sugars intended for storage or long shipment cannot be too strongly enforced, for only in this way can the losses from fermentation be reduced to a minimum.